CHEMICAL ENGINEERING

ESSENTIALS FOR THE CPI PROFESSIONAL

www.chemengonline.com

February 2024

Heat Pumps

Process Control

Reactors

Laboratory Ventilation

Scale Formation

Recycling Equipment

Piloting Distillation Processes

page 24

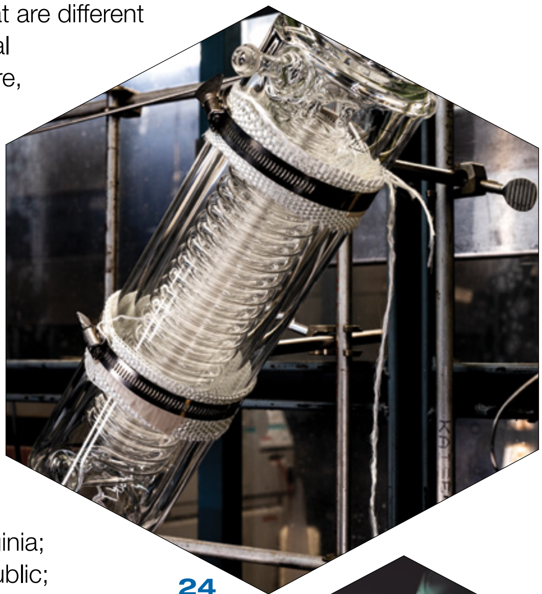
Access Intelligence

CHEMICAL ENGINEERING
ESSENTIALS FOR THE CPI PROFESSIONAL

www.chemengonline.com

February 2024 Volume 131 | no. 2

Cover Story



24

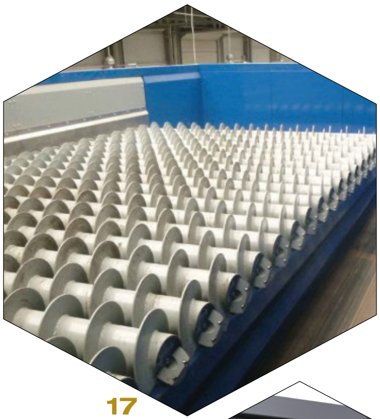
In the News

13

Technical and Practical

31

35



17

19

42

Chemical Connections

Join the *Chemical Engineering* Magazine LinkedIn Group

Visit us on www.chemengonline.com for more articles, Latest News, New Products, Webinars, Test your Knowledge Quizzes, Bookshelf and more

Coming in March

Look for: **Feature Reports** on Weighing; and Flow Measurement and Control; A **Focus** on Pipes, Tubes and Fittings; A **Facts at your Fingertips** on Combustion and Burners; a **Newsfront** on Heat Exchangers; **New Products**; and much more

Cover design:
Tara Bekman
Cover image:
Courtesy of AVN Corp.

CHEMICAL ENGINEERING
ESSENTIALS FOR THE CPI PROFESSIONAL

Editor's Page

Advancing manufacturing 'smartly'

It is an exciting time to be involved in science and technology. Advances across many scientific disciplines are being made at an ever increasing rate. Drivers such as the urgent push toward decarbonization, global competition, the need for medical advances (as seen with the critical need for vaccine development in recent years) and an improved supply chain, along with associated governmental funding are fueling these technological advances.

One of the fastest growing areas that affects all the others, is automation. Advances in data collection and analytics, machine learning, artificial intelligence, robotics, quantum computing and more are enabling new innovations that were not possible even a short time ago. One quick example is additive manufacturing, where customized parts can now be made on demand. It struck me, for instance, in reading our Cover Story on distillation that the authors mention the possibility of fabricating metal trays by additive manufacturing for specialized applications (p. 26). This would not have been possible a relatively short time ago.

Smart manufacturing

The use of advanced and cutting-edge technologies, such as those mentioned above, to improve all aspects of manufacturing is often called "smart" manufacturing. Opportunities exist in increasing efficiency, in helping to meet sustainability goals, in improving the supply chain and much more. While many industrial manufacturers are reaping the benefits of cutting-edge technologies, such progress may be difficult for smaller companies that do not have the resources to do so. A recent report from the National Academies of Sciences, Engineering, and Medicine [*1*] advises that a national effort is needed to advance smart manufacturing in the U.S. The report acknowledges that moving forward with smart manufacturing requires input from a wide breadth of disciplines of science, engineering and social sciences and it recommends that the U.S. Dept. of Energy (DOE) and other federal agencies fund programs to work at the intersections of these disciplines. Specifically, the report identifies six high-demand interdisciplinary technologies where funding is recommended: 1) human-machine co-piloting; 2) sensing; 3) artificial intelligence and machine learning; 4) platforms; 5) digital twins; and 6) uncertainty quantification. The report also recommends education-focused initiatives for the manufacturing workforce, noting that manufacturers cite a lack of a skilled workforce as a leading bottleneck for implementing these emerging technologies.

The National Academies addressed the topic of digital twins in a separate recent report [*2*]. Citing the great potential that digital twins have to accelerate innovation across multiple disciplines, the report outlines recommendations for multiple federal agencies to support development of this technology.

To read how some of the latest technologies are being used in process control, see this month's Newsfront: Process Control: Optimization at the Edge (pp. 13–16). ■

Dorothy Lozowski, Editorial Director

1. National Academies of Sciences, Engineering, and Medicine, Options for a National Plan for Smart Manufacturing, Washington, D.C., The National Academies Press, 2023, https://doi.org/10.17226/27260.
2. National Academies of Sciences, Engineering, and Medicine, Foundational Research Gaps and Future Directions for Digital Twins, Washington, D.C., The National Academies Press, 2023, https://doi.org/10.17226/26894.

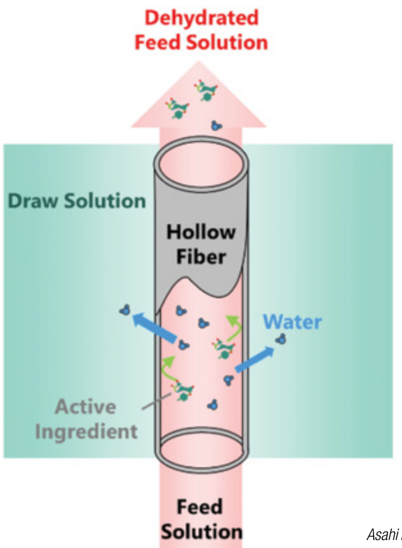
New membranes for dehydrating organic solvents

Researchers at Asahi Kasei Corp. (Düsseldorf, Germany and Tokyo, Japan; www.asahi-kasei.com) are developing a membrane-based system that dehydrates organic solvents without heat or pressure. The system is targeting pharmaceutical-manufacturing applications, where organic solvents are commonly used in performing reactions, as well as for purification by crystallization. For such applications, water needs to be removed from the solvent (dehydration).

Standard dehydration methods, such as vacuum distillation, require the application of heat. These methods are not only time- and energy-intensive, but may also have undesired effects on the pharmaceutical intermediates contained in the organic solution, especially those that are sensitive to heat.

Asahi Kasei has leveraged its expertise to develop this forward-osmosis system, which includes a hollow-fiber membrane module and a draw solution suitable for organic solvents used in pharmaceutical manufacturing. The development of the forward-osmosis polymer membrane and the draw solution enables the removal of water without the loss of pharmaceutical intermediates (diagram). Being able to handle highly soluble liquids, such as tetrahydrofuran (THF), toluene or methanol, this membrane system can dehydrate organic solvents below 1,000 parts per million (ppm) without applying heat or pressure, thereby minimizing the impact on heat-sensitive pharmaceutical intermediates. Compared to vacuum distillation, the process time required can be shortened and energy consumption can be significantly reduced, the company says. The membrane system can be used with a variety of organic solvents, including alcohols, ethers, esters and hydrocarbons.

To evaluate the performance of the membrane system in practical use, Asahi Kasei is collaborating with Ono Pharmaceutical Co. (Osaka, Japan; www.ono-pharma.com), one of the largest pharmaceutical companies in Japan. Commercialization is targeted for 2027.



Asahi Kasei

Pilot plant planned for CO_2 capture project using carbonate fuel cells

ExxonMobil affiliate Esso Nederland B.V. (Rotterdam, the Netherlands; www.esso.nl) is planning to build a pilot plant at its Rotterdam manufacturing complex to generate performance and operability data for a modular carbon-capture and storage (CCS) technology based on carbonate fuel cells (CFC).

The CFC technology, which can generate electricity as it captures CO_2 from exhaust gas, was developed jointly with FuelCell Energy Inc. (Danbury, Conn.; www.fuelcellenergy.com). The company says the project is the first time CFC technology will be piloted for carbon capture in an industrial setting. CO_2 captured from the plant will be transported and permanently stored in empty gas fields under the North Sea as part of the Porthos project (www.porthosco2.nl).

In FuelCell Energy's CFC technology, CO_2-containing exhaust gas is routed into the fuel cells, which capture and concentrate the CO_2 via a side reaction as the fuel cells generate power from fuel. The fuel cells' electrochemical reactions are "supported by an electrolyte layer in which carbonate ions serve as the ion bridge that completes the electrical circuit," the company states.

When CFCs are applied to carbon capture, exhaust gas from an industrial process is fed into the fuel cell's cathode. The CO_2 in the exhaust gas is transferred to the anode side of the fuel cell, where it is much more concentrated and easier to separate, FuelCell Energy says. "During power generation, the carbonate ion transfer results in CO_2 being produced in the fuel electrodes and consumed in the air electrodes," FuelCell Energy says. CO_2 from the anode can be purified by chilling the stream to extract liquid CO_2.

Edited by:
Gerald Ondrey

CO_2 TO METHANOL

Last December, Sumitomo Chemical Co, Ltd. (Tokyo, Japan; www.sumitomo-chem.co.jp) started operating a pilot facility for producing methanol from CO_2 at its Ehime Works, located in Niihama City, Ehime Prefecture, Japan. The company aims to complete the demonstration of this technology by 2028, as well as start commercial production using the new process, and license the technology to other companies in the 2030s. The facility was built with the support of the Green Innovation Fund of the New Energy and Industrial Technology Development Organization.

Conventional CO_2-to-methanol processes have a low yield, due to the reversible nature of the reaction, as well as catalyst degradation caused by byproduct water. Sumitomo Chemical has resolved these issues through joint development with professor Koji Omata of Shimane University Interdisciplinary Faculty of Science and Engineering, leveraging the internal condensation reactor (ICR), a technology that Professor Omata has been developing. The ICR enables the condensation and separation of methanol and water within the reactor. This helps to improve yield, downsize equipment and achieve higher energy efficiency, while it is also expected to prevent catalyst degradation, the company says.

PHOSPHOGYPSUM

Last month, thyssenkrupp Uhde GmbH (Dortmund, Germany; www.thyssenkrupp-uhde.com) signed a master agreement with Ma'aden (Saudi Arabian Mining Co.; Riyadh; www.maaden.

(Continues on p. 6)

com.sa) for the development, engineering and licensing of a calcination plant for phosphogypsum processing. The purpose of the proposed plant, to be located at Ma'aden's Ras al Khair site in Saudi Arabia, will be to recycle phosphogypsum and enable the capture of CO_2 emissions. The joint research and development will be carried out together with thyssenkrupp Polysius and Metso Outotec Oyj (Espoo, Finland; www.metso.com).

Currently, significant amounts of phosphogypsum are produced as a byproduct of phosphoric acid production, which is essential for producing phosphate fertilizers. The options for using phosphogypsum directly are very limited due to impurities and the general properties of this material.

The project will further develop Ma'aden Phosphate's patented technology for reducing CO_2 emissions and recycling phosphogypsum into a useful resource. The phosphogypsum-treatment process converts phosphogypsum into quicklime (CaO). By using alternative fuels, such as hydrogen or sulfur, this calcination step is low in CO_2 emissions. The process also enables the recovery of sulfuric acid, which can be recycled and reused as feedstock for phosphoric acid production. Finally, the quicklime binds CO_2 through a carbonization process to form limestone, which can then be used in the construction industry or for cement production.

METAL MONITORING

The continuous metals-monitoring technology of Sensmet Oy (Oulu, Finland; www.sensmet.com) has recently been evaluated in a field trial conducted by Metso

(Continues on p. 8)

Convert CO_2 to methanol at room temperature

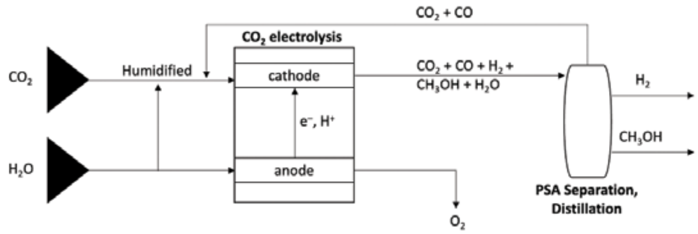
A new electrocatalytic technology (diagram) is said to be the first to produce methanol from CO_2 at ambient temperature and pressure in a commercially viable reactor design. Oxylus Energy (New Haven, Conn.; www.oxylusenergy.com) recently unveiled a 5-cm^2 electrolysis cell to demonstrate its electrified CO_2-conversion process.

Special modifications to the cobalt phthalocyanine catalyst, developed by professor Hailiang Wang and Oxylus chief technology officer and co-founder Conor Rooney at Yale University's Energy Science Institute, enable the process to operate at much milder process conditions, but also significantly increase the methanol selectivity of the reaction. "There are no other catalysts that can do low-temperature and low-pressure CO_2 electrolysis to methanol," emphasizes Perry Bakas, CEO and co-founder of Oxylus Energy. The first iteration of the catalyst involved cobalt phthalocyanine being spread over carbon nanotubes, the addition of a microporous layer to the catalytic electrode structure and the amendment of an amine to the cobalt phthalocyanine's ligands, explains Bakas.

Initial runs showed reaction selectivity for methanol of around 40%, but subsequent kinetic studies have continued to improve upon the Faradaic efficiency of the reaction. The microporous layer greatly enhanced mass transport of CO and increased Faradaic efficiency from 40 to 66%, according to work published last year in *Nature Synthesis*. "We've now achieved well over 80% selectivity of the CO_2-to-methanol conversion. We've really proven that we can do this at a commercially relevant selectivity," says Bakas.

Oxylus is now building membrane-electrode assembly reactors around this catalyst. "The reactor is similar to a typical proton-exchange-membrane (PEM) electrolyzer setup, but on the cathode side, there's a gas-diffusion electrode where the CO_2 conversion takes place," says Bakas. Alongside the launch of the 5-cm^2 cell, the team is currently running tests on a 50-cm^2 cell.

Oxylus



Flow reactor safely performs SuFEx click chemistry

Click chemistry — the subject of the 2022 Nobel Prize in Chemistry — is a powerful and efficient method for rapidly connecting chemical fragments. One highly versatile type of click chemistry involves the so-called SuFEx [sulfur(VI) fluoride exchange] reaction, which results in the formation of covalent bonds under mild conditions and can greatly simplify the synthesis of therapeutically relevant small molecules, peptides and proteins. SuFEx chemistry involves attaching a $–SO_2F$ molecular moiety, after which the F atom can easily be replaced with a range of functional molecular groups of therapeutic value. An obvious reagent for introducing the $–SO_2F$ moiety is the gaseous component sulfuryl fluoride SO_2F_2, but since this is mildly toxic, it requires elaborate synthetic procedures to avoid its direct use.

Now, researchers at the Flow Chemistry group of the University of Amsterdam's Van 't Hoff Institute for Molecular Sciences (the Netherlands; (hims.uva.nl) have developed a modular flow-chemistry platform for a safe and efficient execution of SuFEx click chemistry. In a recent article in *Nature Synthesis*, they describe how their platform generates the toxic gaseous SO_2F_2 reagent in a safe and controlled manner, and how it facilitates the subsequent fast and selective functionalization of small molecules, peptides and proteins for therapeutic purposes.

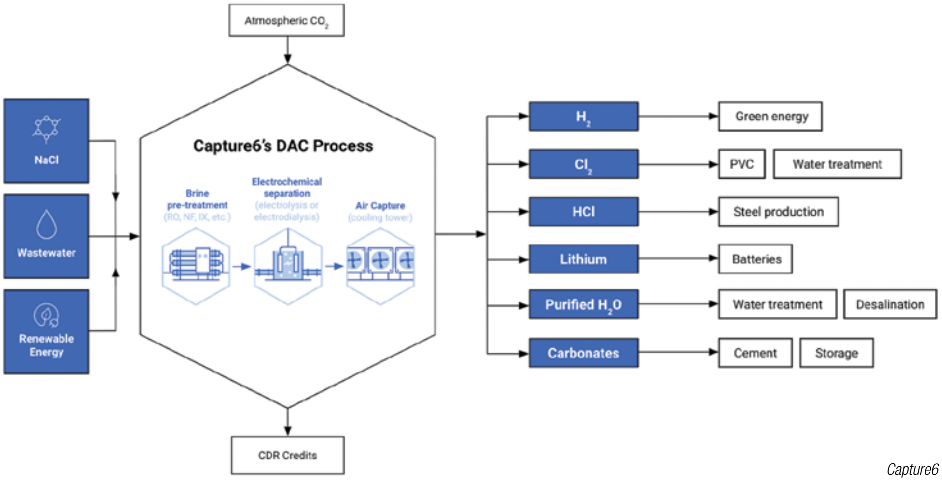
The system consists of two interconnected flow reactors. The first generates SO_2F_2 in a controlled and dosed manner from inexpensive commodity chemicals sulfuryl chloride (SO_2Cl_2) and potassium fluoride. In the second reactor, the generated gaseous SO_2F_2 is mixed with other reactants, ultimately yielding the desired SuFEx product. By immediately reacting away the SO_2F_2, the modular system effectively eliminates the safety and practical concerns of the toxic reactant. While exploring the performance of their system, the researchers were able to obtain a diverse range of SuFEx products with excellent yields in just two minutes of residence time. They attribute this short residence time to the intimate contact between gas and liquid phase in a flow system.

This process makes freshwater from brine while capturing CO_2

A new electrochemical technology for direct-air capture (DAC) of atmospheric CO_2 can also produce freshwater from brackish water or brine waste from desalination or wastewater treatment facilities (diagram). The technology, developed by Capture6 (Berkeley, Calif.; www.capture6.org), splits the salt content from brine waste, forming an acid and a base. The derived base acts as a solvent, absorbing CO_2 from the air and forming carbonates that can be sequestered. "By consuming salt from wastewater, the technology generates the solvent for DAC, while simultaneously producing freshwater," explains Capture6's chief technology officer Rahul Surana. According to the company, the process can recover over 50% of freshwater from desalination waste brine, and it can be configured to handle varying saltwater sources and to produce different byproducts.

This DAC process has several features that contribute to its scalability. First, it operates at ambient temperatures, making it compatible with renewable-energy sources, and it is a once-through process, meaning that fresh solvent is continuously created, says Surana. Also, the process comprises multiple technical components that are widely used in industry, such as nanofiltration and electrodialysis. Notably, the process' air contactors expand upon traditional cooling-tower designs with Capture6's AURA (Absorption Reactor Unit for Ambient Air) technology, for which the company was recently awarded funding from the U.S. Dept. of Energy. "This enhanced tower design will likely improve upon the mass-transfer characteristics of the process and reduce total energy demand. With the award, we expect to test various configurations that combine the laminar airflow in the cooling tower with turbulent airflows and atomize the liquid solvent as in a venturi," says Surana. Optimizing AURA is expected to improve the efficiency and the energy intensity of the DAC process. The company estimates AURA can reduce the total energy required for DAC by 15% when compared to other solvent-based DAC technologies.

Capture6 is working with the Palmdale Water District in California on the Project Monarch pilot plant, which will be the first fully integrated water-management and CO_2-removal facility of its kind. Groundbreaking for Project Monarch is expected in the second quarter of 2024.



Capture6

at its facilities in Pori, Finland. The trial compared lithium and sodium measurements by laboratory ICP-OES analysis (inductively coupled plasma, optical-emission spectroscopy) with the Sensmet μDOES continuous analyzer, and demonstrated "excellent" correlation between the two different methods, the company says.

During the trial, samples were taken from a process in which battery-grade lithium hydroxide monohydrate was produced from spodumene concentrate treated by high-temperature conversion in a rotary kiln. The

(Continues on p. 9)

Reducing CO_2 emissions from pig iron production

Researchers of Karlsruhe Institute of Technology (KIT; Germany; www.kit.edu) and the SMS group GmbH (Düsseldorf, Germany; www.sms-group.com) have developed a new process to reduce CO_2 emissions from worldwide steel production by several hundred million tons per year. It is based on modernizing blast-furnace technology with moderate investments.

The process was developed by KIT's Institute for Chemical Technology and Polymer Chemistry (ITCP), in cooperation with the SMS group and its subsidiary Paul Wurth S.A. (Luxembourg; www.paulwurth.com), and KIT's startup omegadot software & consulting GmbH (Mannheim, Germany; omegadot.software). The process was demonstrated and validated at a pilot plant at Dillinger Hüttenwerke, Saarland, which is operated by the SMS group, together with Dillinger Hüttenwerke and Saarstahl. The study, described in a recent issue of *Energy Advances*, was performed in cooperation with omegadot, which developed a software for the precise simulation and visualization of the process and for supporting scale-up to an industrial plant.

In the process, a mixture of coke-oven gas (COG) and blast-furnace gas (BFG) is heated to high temperatures in a regenerative heat exchanger, and undergoes dry reforming into synthesis gas (syngas; $CO + H_2$). The syngas can be injected into the blast furnace, where it serves as the reducing agent instead of coke in iron production. "Per ton of steel produced, significant amounts of coke can be saved," says ITCP's Philipp Blanck, who cooperated closely with the SMS group at the pilot plant that was part of the steelworks. "Specific CO_2 emissions are reduced by up to 12%," he says.

"Integration of the new process in the steelworks is the first step in the transformation of steel industry," says Gilles Kass from the Research Section of SMS group and a coauthor of the publication.

Recycle any type of magnet from end-of-life products

A new pilot plant is demonstrating a proprietary technology for recycling magnet materials from end-of-life (EOL) products, such as electric motors, automobile parts and hard-disk drives. Developed by Cyclic Materials Inc. (Toronto, Ont., Canada; www.cyclicmaterials.earth), the Mag-Xtract process is said to be the only magnet-recycling technology on the market that can isolate magnets at any cleanliness level from any kind of EOL product at scale. Being agnostic to magnet type is a significant breakthrough, says Cyclic Materials' CEO Ahmad Ghahreman, because magnet feedstock rarely contains just one type of magnet — typically it is a mixture of four different types of magnetic materials, and each magnetic material comes with many different magnet grades. The Mag-Xtract technology can be combined with Cyclic Materials' proprietary hydrometallurgical technology to process recycled magnet materials and other manufacturing waste to produce mixed raw materials, including rare-earth oxides and other byproducts that can be repurposed for new applications. According to the company, this is the first magnet-agnostic process ever that can yield mixed rare-earth oxides from recycled products.

In December, the company opened its Mag-Xtract pilot plant in Kingston, Ont., which is designed to process 8,000 tons/yr of magnet-containing products. Initial runs at the plant have processed several tons of feedstock per day. "Last fall, Cyclic Materials also demonstrated the capacity to recycle 10 ton/yr of magnet feedstock and manufacturing waste with its hydrometallurgical technology, which the company is currently scaling," adds Ghahreman. The first commercial demonstration plant for this technology is slated to launch in the second quarter of 2024. Besides mixed rare-earth oxides and copper, which are the main products, the recycling technologies can also produce several other products, including aluminum, steel, cobalt and nickel hydroxides and boron.

"Future company plans involve creating integrated plants using Mag-Xtract to process magnet-containing feedstock for a central hydrometallurgical processing plant, which will take in recycled magnet feedstock and manufacturing waste to produce many valuable raw materials," says Ghahreman.

hydrometallurgical technology, developed by Metso, produces battery-grade $LiOH{\cdot}H_2O$ by soda pressure leaching. In this application, the analytical performance of the μDOES analyzer was evaluated for the continuous optimization of pressure leaching and conversion leaching.

Following the trial, Metso will adopt μDOES technology in selected Courier HX systems for Li and Na analysis, and the new systems will be available globally in 2024.

Sensmet's patented μDOES technology is based on atomic

(Continues on p. 10)

emission spectroscopy. A microdischarge is created directly inside the aqueous sample, causing a tiny volume of the fluid to be flash-heated to 10,000°C. Molecular species in the microdischarge are dissociated into atoms, which are excited to their respective higher electronic states. Upon returning to their ground state, these atoms release their excess energy by emitting light at their characteristic wavelengths. The µDOES measures this atomic emission spectrum to derive quantitative analysis of the metals contained in the sample.

DECARBONIZING

At Nestlé's plant in Nunspeet, the Netherlands, GEA AG (Düsseldorf, Germany: www.gea.com) is to equip a milk-powder line for hypoallergenic-infant formula with process and heat-pump technology. Using heat recovery from the spray dryer and further processes, the plant will use 75% less energy for steam and massively reduce carbon emissions, GEA says. ❒

Copper-graphene composite could enable more efficient electrical hardware

Composites of copper and graphene have been explored as a way to improve electrical conductivity, but researchers have not been successful at enhancing conductivity with a corresponding reduction in the temperature coefficient of resistance (TCR), and have not been able to find ways to make the composites at larger scales. The ability to synthesize enhanced-conductivity, low-TCR materials at scale could mean more efficient electrical machines and grid transmission. Scientists at Pacific Northwest National Laboratory (PNNL; Richland, Wash.; www.pnnl.gov) have taken a step in that direction by synthesizing copper-graphene composites using a PNNL-developed solids process known as shear-assisted processing and extrusion (ShAPE). The ShAPE process combines heating and physical deformation of the metal, and has been used to reduce costs for making high-strength aluminum alloys (*Chem. Eng.*, September 2022, p. 11).

By adding 18 parts per million (ppm) graphene to copper 11000 alloy (a widely used metal for current-carrying hardware) using ShAPE, the PNNL researchers produced composites with an 11% reduction in TCR with improved conductivity in bulk-scale wires (1.5 mm dia.; 0.2-m length).

The ShAPE solids-processing technique used to extrude the composite wire leads to a "uniform, near pore-free microstructure, punctuated with tiny flakes and clusters of graphene that may be responsible for decreasing coefficient of resistance of the composite," the PNNL team says. Both flakes and clusters must be present to make better conductors for high-temperature operations, they add.

The study "demonstrates the potential of ShAPE for producing bulk-scale industrially viable [copper-graphene] composites with lower TCR, as well as improved electrical conductivity," PNNL says. The work was published in the January 2024 issue of *Materials & Design*. ■

Business News

Plant Watch

Solenis to build new polyvinylamine production facility in Virginia

January 12, 2024 — Solenis, LLC (Wilmington, Del.; www.solenis.com) will invest $193 million to expand production capacity of polyvinylamine (PVAm) in Suffolk, Va. The company will build a new 80,000-ft^2 production unit, packaging facility and tank farm for PVAm products, which are used in paper and cardboard manufacturing. This new facility will be used to expand the existing polymer product line and add additional capacity of this critical monomer.

Wacker to build new silicones production site in Czech Republic

January 11, 2024 — Wacker Chemie AG (Munich, Germany; www.wacker.com) plans to build a new production site for silicones in Karlovy Vary, Czech Republic. Production is expected to start at the end of 2025. The new production site in Karlovy Vary is intended to complement Wacker's existing integrated production sites in Germany (Burghausen and Nünchritz), as well as a site in Pilsen, Czech Republic. When fully operational, the new site will be able to supply over 20,000 metric tons per year (m.t./yr) of custom-made silicones.

Nouryon completes colloidal silica capacity expansion in Wisconsin

January 10, 2024 — Nouryon (Amsterdam, the Netherlands; www.nouyron.com) has expanded its production capacity for Levasil colloidal silica products by nearly 50% at its manufacturing facility in Green Bay, Wis. The colloidal silica products made at the site can be used in electronics and building and construction applications.

AGC Biologics to build one of Japan's largest biomanufacturing sites

January 5, 2024 — AGC Biologics Inc. (Bothell, Wash.; www.agcbio.com) plans to construct a new manufacturing facility at AGC Inc.'s Yokohama Technical Center in Japan. The facility is expected to be operational in 2026. The site will house multiple 2,000-L single-use bioreactors and several 4,000-L or larger reactors for mammalian-cell-culture services, making it one of Japan's largest sites for this type of manufacturing, says AGC.

Metso to build ferrochrome plant for FACOR in India

January 3, 2024 — Metso Corp. (Espoo, Finland; www.metso.com) has agreed to build a new ferrochrome plant for Ferro Alloys Corp.'s (FACOR) expansion project in Bhadrak, Odisha, in India. The new plant will produce 300,000 m.t./yr of ferrochrome, and it is anticipated to start operating in 2025.

LG Chem begins construction of major battery-materials plant in Tennessee

December 21, 2023 — LG Chem Ltd. (Seoul, South Korea; www.lgchem.com) commenced the construction of a cathode plant in Clarksville, Tenn. LG Chem will invest around KRW 2 trillion ($1.6 billion) in the first phase of construction to build a plant with a capacity of 60,000 m.t./yr of NCMA (nickel-cobalt-manganese-aluminum) cathode materials. The plant is expected to be the largest cathode-material facility in the U.S. when production begins in 2026.

Idemitsu Kosan starts up production plant in Malaysia for syndiotactic polystyrene

December 19, 2023 — Idemitsu Kosan Co. (Tokyo; www.idemitsu.com) has constructed a second production plant for syndiotactic polystyrene (SPS) in Pasir Gudang, Johor, Malaysia. The production capacity of this new plant is 9,000 m.t./yr, effectively doubling SPS production at the site. SPS resin is widely used in automotive parts and home appliances.

Air Liquide to build new carbon-capture plant in Rotterdam

December 19, 2023 — Air Liquide S.A. (Paris, France; www.airliquide.com) will build, own and operate a world-scale carbon-capture unit at its hydrogen production plant in the Port of Rotterdam. The carbon capture unit will be operational in 2026. Through this project, Air Liquide will be able to supply hydrogen that is significantly decarbonized.

Ineos starts up Europe's largest cumene plant in Germany

December 18, 2023 — Ineos Ltd. (London, U.K.; www.ineos.com) announced that Ineos Phenol started production at Europe's largest cumene facility in Marl, Germany. The 750,000-m.t./yr facility will use existing pipeline connections between Ineos' phenol and acetone production sites in Gladbeck, the Evonik Chempark in Marl, and the BP petroleum refinery and cracker complex in Gelsenkirchen. Cumene is used in the production of phenol and acetone, which are used in many medical and personal-care applications, including aspirin, lozenges, contact lenses and more.

Evonik to increase production capacities for hydroxyl-terminated polybutadienes

December 15, 2023 — Evonik Industries AG (Essen, Germany; www.evonik.com) has begun work to expand its hydroxyl-terminated polybutadienes (HTPB) plant in Marl, Germany. Evonik's Coating & Adhesive Resins division expects the additional production capacity for its HTPB products, marketed under the Polyvest HT brand, to be available as early as the second quarter of 2024.

LINEUP

Look for more latest news on chemengonline.com

Mergers & Acquisitions

Mitsui-Celanese JV begins producing low-carbon methanol

January 15, 2024 — Fairway Methanol LLC, a 50-50 joint venture (JV) between Mitsui & Co., Ltd. (Tokyo; www.mitsui.com) and Celanese Corp. (Dallas, Tex.; www.celanese.com), has begun the production of methanol using CO_2 emitted from plants surrounding the JV facility in Clear Lake, Tex. Fairway Methanol is expected to capture 180,000 m.t./yr of CO_2 and produce 130,000 m.t./yr of low-carbon methanol.

UPL to acquire fungicide business from Corteva

January 11, 2024 — UPL Europe Ltd. (Warrington, U.K.; www.upl-ltd.com), a global provider of agricultural products, announced the planned acquisition of Corteva Agriscience's (Indianapolis, Ind.; www.corteva.com) solo mancozeb global fungicide business outside of China, Japan, South Korea and E.U. member countries. The acquisition will give UPL ownership of Dithane-branded mancozeb products, as well as access to the Rainshield technology, which enables crop protection in wet weather conditions. Mancozeb is a highly effective protective fungicide used to prevent plant diseases across a range of crops, including rice, soybean, wheat, onions, potatoes and other vegetables and fruits.

Altana to acquire U.S.-based pigments company

January 10, 2024 — Altana AG (Wesel, Germany; www.altana.com) has agreed to acquire the Silberline Group., a Pennsylvania-based company specialized in effect pigments utilized in various applications, ranging from automotive coatings and printing inks to plastics, protective coatings and packaged consumer goods. In 2022, Silberline reported sales of around $80 million.

LanzaTech and Tadweer explore transforming solid waste into SAF

December 18, 2023 — LanzaTech Global, Inc. (Skokie, Ill.; www.lanzatech.com) and Tadweer Waste Treatment LLC (Abu Dhabi, U.A.E.; www.tadweer.com) have joined forces to initiate an integrated feasibility study to scale up the production of sustainable aviation fuel (SAF) from municipal and commercial solid waste. The partners anticipate that up to 350,000 m.t./yr of hard-to-recycle municipal and commercial solid waste could be transformed into 200,000 m.t./yr of ethanol, ultimately producing 120,000 m.t./yr of SAF.

ADNOC to acquire OCI's stake in Fertiglobe

December 18, 2023 — ADNOC (Abu Dhabi, U.A.E.; www.adnoc.ae) and OCI Global N.V. (Amsterdam, the Netherlands; www.oci-global.com) announced the acquisition by ADNOC of OCI's entire majority shareholding in Fertiglobe plc. Fertiglobe is the world's largest seaborne exporter of urea and ammonia combined, and the largest nitrogen fertilizer producer in the Middle East and North Africa, with production facilities in Egypt, Algeria and the U.A.E. Under this agreement, ADNOC will purchase OCI's stake in Fertiglobe for AED 13.28 billion ($3.62 billion). ■

Mary Page Bailey

Process Control: Optimization at the Edge

The key technologies shaping industrial process control all work in tandem to support the ultimate goal of plant optimization

Process control technologies are at the heart of all chemical process facilities, providing the foundation for safe and efficient plants. As traditional process control systems are evolving to integrate more automated elements, including artificial intelligence (AI), edge connectivity, faster communications and "smarter" field devices and sensors, plants can reach new levels of optimization on the path toward more autonomous operations (Figure 1).

FIGURE 1. The advancement of process control involves integrating the physical control devices with digital innovations via cloud and edge connectivity, sophisticated data analytics and artificial intelligence

Defining autonomy

"Autonomy is a part of a larger topic, which is the optimization of assets. When we talked about optimization in the past, it was about getting better sensors with higher accuracy and repeatability, but when we talk about optimization today, including the journey to autonomy, it's more about generating the right data and how to make that data available in a way that can actually be used to further optimize operations," says Claudio Fayad, vice president of technology, process systems and solutions at Emerson Co. (St. Louis, Mo.; www.emerson.com).

Kevin Finnan, advisor of market intelligence and strategy at Yokogawa Corp. of America (Sugar Land, Tex.; www.yokogawa.com/us) defines autonomous operations as "the state in which assets and operations throughout possess learning and adaptive capabilities that enable responses with minimal human interaction, thus empowering operators to perform higher-level optimization tasks." In theory, with a well-designed autonomous control platform, operators and subject-matter experts would not even need to be present at a production facility and could instead reside at an integrated operations center serving multiple facilities globally.

Yokogawa has executed several proof-of-concept projects for fully autonomous operations. In 2022, the company collaborated with Eneos Materials to launch a field test using a reinforcement-learning AI platform to autonomously operate a chemical manufacturing plant for 35 days. "This was a world-first project. The test confirmed that reinforcement-learning AI could be safely applied in an actual plant and demonstrated that the technology can control operations that had been beyond the capabilities of other control technologies. Until now, such tasks have necessitated the manual operation of control valves based on the judgment of plant operators," says Finnan. Since the initial test in 2022, the application has now run successfully for over a year and has been officially adopted by Eneos at the facility. For more information on this project, see Autonomous Control of a Distillation Column, *Chem. Eng.*, July 2023, p. 10.

Finnan also cites examples where AI-based asset-management can be used to replace traditional process control technologies, such as proportional–integral–derivative (PID) loops, to improve energy efficiency. For instance, Yokogawa offers a holistic control system to manage fired assets, such as boilers and furnaces, to increase fuel efficiency, extend asset life and minimize downtime. "The PID control in this platform could be replaced by reinforcement-learning AI technology to add additional stability that conserves more energy," he notes.

Next-generation networks

In another proof-of-concept, Yokogawa collaborated with NTT Docomo to test an autonomous AI and cloud-based control solution, along with a 5G mobile communications network. The test successfully controlled a simulated process plant operation and demonstrated that 5G is suitable for remote control of actual plant processes. "In conjunction with AI and cloud, 5G communication, which offers low latency and the capability to connect numerous devices, promises to be a core technology for autonomous operations. Compared

FIGURE 2. For plants to fully take advantage of the faster response enabled by 5G communication, it is crucial that mobile communication devices be designed for hazardous-area use

to 4G communication, the test demonstrated that, especially with high-speed control, 5G provides lower latency and less overshoot relative to the target," says Finnan. He also mentions that 5G can handle control cycles as short as 0.2 s, providing faster process control that can result in higher product quality and energy efficiency.

A partnership between Nokia Corp. (Espoo, Finland; www.nokia.com) and i.safe Mobile GmbH (Lauda-Koenigshofen, Germany; www.isafe-mobile.com) will see i.safe Mobile's IS540.x intrinsically safe smartphone — said to be the world's first 5G-enabled smartphone for hazardous-area use — integrated with Nokia's end-to-end Nokia Digital Automation Cloud (DAC) platform, as well as the company's private wireless infrastructure and edge applications. "In hazardous areas, it is vital that workers are connected using devices that are Ex certified. As a result of this international partnership, Nokia will extend its industrial device portfolio with our products to support the connectivity needs of organizations in the chemical, pharmaceutical and mining sectors," according to i.safe Mobile. The company emphasizes the benefits of industrial 5G communication, including the secure integration and consolidation of complex workflows. The company is also developing the industry's first 5G-enabled Android and Windows tablets (Figure 2) for Zone 1/21 with charging options for hazardous areas, which are expected to be available later in 2024.

Data in context

It is well-known across industry that the usefulness of process data is limited if there is little or no context given. Because of the way that legacy distributed control systems (DCS) have been created, there are often many kinds of independent systems where the data and its associated models may only exist within that system, with no context linking it to other parts of the plant. "These systems are very good at transferring data like temperature or flow, but they are not very good at transferring the meaning of the data. So even when you build a system of systems, to try to bring the data together, it doesn't include the context that allows an autonomous system to make sense of the data," says Fayad. One way that plants can move away from this "system of systems" approach is with an integrated control platform that includes edge connectivity. The main principle of edge computing is to bring data from disparate sources closer to the actual points where it will be processed.

Emerson recently launched its DeltaV Edge Environment (Figure 3), which replicates the data gathered from the DCS and makes it available for other applications, such as data analysis or optimization platforms. "The Edge Environment is connected to the rest of the DeltaV control system, so there is only a unidirectional path for the data from the controllers to the Edge Environment. People can actually view and manipulate the data with context, making queries about the status of the plant or feeding a digital twin, without actually needing any physical access to the controllers and the critical parts of the DeltaV system," says Fayad. While obviously promoting improved data utilization, this setup is also a boon for plant security, since it provides broader access to DCS data without expanding access to the control system itself.

Another facet of next-generation control systems is the integration of AI and machine learning. Through Emerson's partnership with Aspen Technology, Inc. (Bedford, Mass. www.aspentech.com), the MTell platform helps to support advanced automation through AI-supported anomaly detection. "Models are created to understand how a certain process needs to operate, and if something goes wrong, they can identify when the process is out of its operating envelope. If we can get enough data in a secure way to have a digital twin and they can check if the plant is operating with the right parameters, and adjusting setpoints as needed, that is how we close the loop to the autonomous operation," explains Fayad.

As part of their process-control objectives, many manufacturers are implementing overall equipment effectiveness (OEE) analytics, which can quantify production losses due to availability, performance and quality. Such process-control improvement can be the key to enhanced process performance that can maintain operations at target rates, even in the face of process upsets and quality-related OEE losses, says John Cox, principal analytics engineer at Seeq Corp. (Seattle, Wash.; www.seeq.com).

Being able to intelligently analyze, identify and respond to upset conditions is a major step toward the data contextualization required for more autonomous operations. "Analytics insights drive autonomous operation through actionable monitoring and diagnostics focused on process-control performance and service factors, and crew-by-crew interactions with the regulatory control layer," explains Cox. Once implemented, these monitoring and diagnostics capabilities can be easily scaled across controllers, control valves and similar processing equipment to help rapidly identify and prioritize optimization opportunities. "Analytics can be used to contextualize everything from leaky control valves to site-wide carbon intensity, aggregated by product and energy or utility streams. Process-control expertise can transform these insights into process-control improvements," says Cox.

Cloud-based operations

Cloud computing technologies are making vast amounts of process data more accessible than ever before, helping engineers to make better-informed choices. "Many products are contributing toward fully automated industrial operations. Cloud-based platforms in particular enhance centralized control and data analysis, thus improving decision-making in autonomous operations. For instance, cloud solutions streamline engineering and simulation in automation, while intelligent analysis of field-device data aids in optimizing plants," says Rebecca Vangenechten, head of automation and engineering, process industries, at Siemens AG (Munich, Germany; www.siemens.com). This unprecedented access to data means that engineers from any location can connect seamlessly and work on several projects at the same time. "This is where the advantages of a web-based control system like Simatic PCS neo come into play. This system facilitates cross-regional and cross-functional collaboration, allowing rapid development of new systems," says Vangenechten.

Siemens recently demonstrated this global reach in a partnership with Dow (Midland, Mich.; www.dow.com) at MXC, an advanced manufacturing institute and innovation center in Chicago, Ill., with a new test bed for the process industries equipped with Simatic PCS neo to exhibit the efficiencies of using a uniform source for engineering data across different disciplines. "When such a setup becomes part of the digital twin technology for production, plant engineers get instant access to live process data, up-to-date plant drawings and documentation. The maintenance aspect of engineering has also benefited from these advancements, as it has led to the creation of digital work orders, significantly reducing paperwork and minimizing confusion. This shift can potentially reduce engineering time by approximately 30%," adds Vangenechten.

Interoperability

Also at the forefront of next-generation process-control technologies is a greater focus on interoperability and open process automation (OPA). "Many chemical companies have found that closed systems are expensive to upgrade and maintain. With proprietary systems, it also becomes more challenging to integrate new technologies. Digital transformations demand enterprise-wide operability, along with cybersecurity, agility and sustainability," notes Yokogawa's Finnan. The OPA Forum (www.opengroup.org) began work in 2016 to develop process automation standards that aim to ensure the adoption of truly interoperable automation systems while providing built-in security, future-proof updates and simplified migration. "These standards will allow users to select 'best-of-breed' software, hardware and other technologies from multiple suppliers and realize much more value from their operations. There are currently several OPA proof-of-concept tests in process," says Finnan. For more on interoperability and OPA stan-

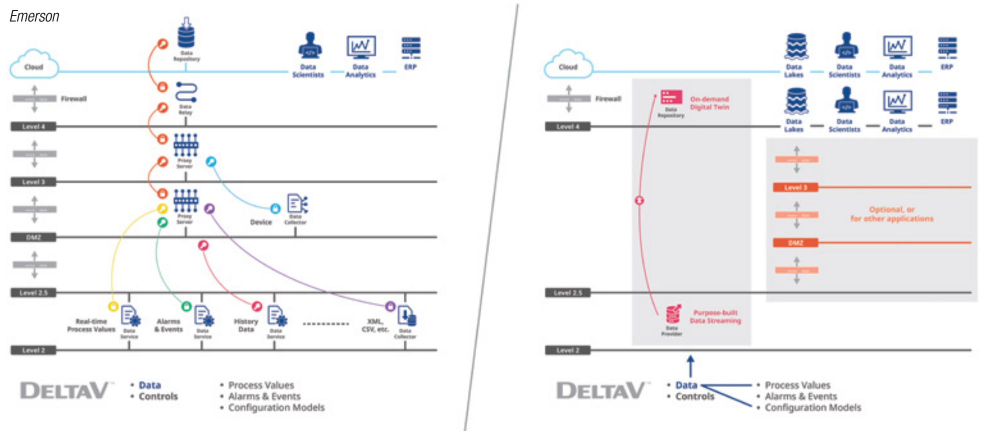


FIGURE 3. The DeltaV Edge Environment consolidates DSC data into a single outbound, unidirectional connection to maximize accessibility to secure, real-time contextualized data

dards, see the two-part Cover Story from the June 2023 issue of *Chem. Eng*, pp. 24–30.

Furthermore, adds Siemens' Vangenechten: "Increased interoperability and standardization allow for enhanced compatibility between different systems and brands, facilitating more seamless operations across various platforms." Interconnection between digital systems is just the beginning, though. "The seamless integration of the physical and virtual worlds will continue to be

FIGURE 4. Communication must be fast and reliable for plants to fully take advantage of the data collected by field devices. Gateway devices can convert and store data from wireless field devices, providing capabilities for remote monitoring and diagnostics

a key factor in designing distributed control systems more efficiently and operating them even more sustainably in the future," she notes. And the benefits of next-generation control systems are not limited to brand-new facilities. "Collaborative development of AI applications facilitates proactive maintenance, and also supports digitizing of documentation for older facilities, laying the groundwork for more efficient, targeted optimization and modernization planning."

Field devices support insight

Beyond the software and fully digital elements of control systems, the physical devices that provide process data to control systems are also undergoing a rapid evolution to meet new demands for process automation and optimization. "Field sensor technology is quickly progressing to more sophisticated, wireless and faster communication protocols," says Kris Worfe, industry marketing manager — chemical, Endress+Hauser USA (Greenwood, Ind.; www.us.endress.com). Wireless gateway technologies, such as the FieldGate SWG50 (Figure 4), are designed to provide secure communication from field devices and help users to monitor measurement and device health status.

"On the horizon are internal improvements to physical devices to include advanced diagnostic and health functions, more intuitive information and data sharing. Field devices are continuously trending in the 'smarter' direction, becoming more capable of sharing their health status and providing process insights," explains Worfe.

But as a foundational step for remote access to the data from these advanced field devices, cloud and edge technologies remain key. "Cloud-based infrastructure supports storing, analyzing and sharing the vast amounts of data generated across the process industries. Interconnected devices and edge computing capabilities are what allow for real-time data processing, enabling faster decision-making and reducing latency in autonomous operations," says Worfe. However, he points out that the adoption of interconnected sensor networks can be a slower process for some organizations, as it may require a change in company culture. "A challenge for end users in the industry could be aligning with internal IT personnel and infrastructure with new features and capabilities."

Sustainable practices

One area where many company cultures are indeed aligning with advances in process control is in the capabilities to improve upon environmental sustainability. "Advanced control systems are optimizing energy consumption by regulating machinery and processes according to real-time demand, reducing unnecessary energy usage," explains Worfe. This optimization of resource consumption stretches beyond energy to other essential parts of a process, including raw materials and water. "Precise control over production processes minimizes material wastage, ensuring materials are used efficiently, consequently reducing the overall environmental impact associated with waste disposal," adds Worfe. He highlights water management as a particular application that can benefit from the remote-monitoring capabilities and waste-reduction realized by modern control systems. "Integrating eco-friendly practices into process control to reduce environmental impact is mainly driven at the corporate level with 'green' or 'drive-to-zero' initiatives," he notes. ■

Mary Page Bailey

Mechanical Recycling

Sort plastic flakes with this machine

The new Innosort Flake (photo) is a high-throughput system for sorting plastic flakes. Enhanced features enable simultaneous flake sorting by polymer type, color and transparency, achieving unmatched quality, even from highly contaminated inputs, the company says. Thanks to its advanced near-infrared (NIR) spectrometer, the machine precisely detects various polymers, allowing for the recovery of recyclable materials from highly contaminated infeed. With this technology, plastics recovered from mixed waste, for example, can be sorted for recycling, giving access to more recyclable materials that otherwise would be lost or downcycled for lower-grade applications. — *Tomra Systems ASA, Asker, Norway*
www.tomra.com

A conveyor for high-speed sorting

The PX acceleration conveyor (photo) meets the demand for higher throughput in sensor-based automatic sorting, which can operate at higher working speeds. To achieve the desired result, this company has integrated the best features of its BB and DB conveyors, and introduced improvements. These include a new air stabilizer, which ensures consistent sorting quality at faster speeds with light materials, resulting in excellent purity of the output. The PX conveyor features a slot to fit a sensor under the belt. It offers a belt speed ranging from 3.2 to 4.5 m/s, and can be specified with two motors to ensure the necessary torque at the required speed. — *Stadler Anlagenbau GmbH, Altshausen, Germany*
https://w-stadler.de

Recycling PET fiber back to PET fiber

Intarema FibrePro:IV (photo) has been developed for fiber-to-fiber recycling of polyethylene terephthalate (PET). Thanks to its especially gentle material preparation and efficient removal of spinning oils, the rPET produced can be reused in proportions of up to 100% for the production of very fine fibers. By combining proven Intarema technology with a new IV optimizer, the process succeeds in processing shredded PET fiber materials heavily contaminated by spinning oils in such a way that the finest fibers can be produced again from the recycled pellets. The system is characterized by a longer residence time of the PET melt — essential for achieving high-quality recycled pellets. — *Erema Group, Ansfelden, Austria*
www.erema.com

Recycling refrigerators in one step

This company is supplying a plant to recycle refrigerators for Enva Northern Ireland Ltd. for its site in Toomebridge. When it starts up this year, the plant will process 70 refrigerators per hour. The heart of the plant is this company's ADuro QZ shredder (photo), which does not use any cutting tools. Instead, it gently and quickly breaks up the input material by using the effects of impact forces. It is a breakthrough technology because it makes valuable waste material quickly accessible without potentially harmful substances emanating from individual parts — all at low operating costs, the company says. — *Andritz AG, Graz, Austria*
www.andritz.com

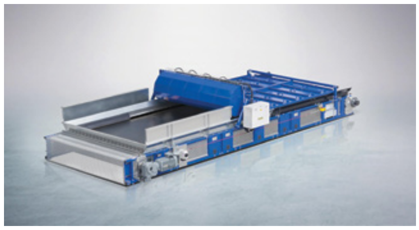
Patented screen for materials-recovery plants

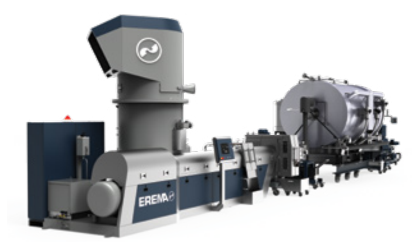
Two years ago, this company partnered with Günther – a German manufacturer specializing in screening technology — to continue supplying world-class recycling solutions to the North American market. Günther's patented Splitter spiral-shaft screen (photo) is a primary separation device for sizing materials at the front end of materials recovery facility (MRF) systems. The screen is highly customizable in size and application and operates effectively at high capacities. Its open-ended design is non-wrapping, self-cleaning and very low maintenance. — *Van Dyk Recycling Solutions, Norwalk, Conn.*
www.vdrs.com

Safe conveying and feeding for black mass recycling

Proper processing of the active material plays an important role in battery

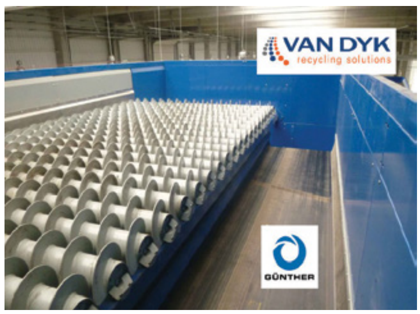
Tomra Systems


Stadler Anlagenbau



Erma Group


Andritz



Van Dyk Recycling Solutions

compound manufacturing. The active material is manufactured in a multistep process that relies upon both safe handling and accurate feeding of bulk materials and additives. This company's conveying systems and feeders, are used for the safe transport and high accuracy in feeding ingredients. The same holds true for black-mass recycling. Due to the use of hazardous materials in active cathode material manufacturing, as well as in black mass recycling, secure containment is essential. The conveying components feature dust-proof design and gentle handling of abrasive bulk materials. High-accuracy, reliable feeders (photo) feature state-of-the-art weighing and control technology. — *Coperion K-Tron, Niederlenz, Switzerland*
www.coperion.com


Coperion K-Tron

Measure and sort challenging black plastic

This company has developed a process for sorting plastic pieces based on material. The company's tracking-type Raman spectroscopy technology enables the identification of materials with high accuracy in a streamlined process, even when sorting plastic pieces of black shades, as well as black plastic pieces mixed with those of other colors, which have thus far been difficult to identify using traditional near infrared (NIR) spectroscopy. A key advantage of the tracking-type Raman spectroscopy technology is that it uses this company's measurement and control devices, so the exact position of plastic pieces on high-speed conveyor belts can be detected. The company plans to market a plastic sorting equipment using this method during the first half of this year. — *Canon Inc., Tokyo, Japan*
https://global.canon

An AI-powered food-grade plastic-sorting project

The OMNI project, directed by this company, Valorplast and TotalEnergies, aims to enhance the circularity of polypropylene (PP) food packaging by leveraging artificial intelligence (AI) and machine learning (ML) for the identification and separation of food-grade PP from household post-consumer wastes. The new technology is based on AI and computer vision, coupled with an efficient decontamination process, to provide a high-performing, marketable solution to tackle the challenge of mechanically recycling PP for food-contact applications. After 18 months of research, OMNI led to an alternative to digital and physical marking solutions, which require system-wide packaging changes. A demonstration unit was built and an AI model trained, based on wastes collected from five locations across France. The AI and robotic sorting achieved a successful pick rate of 50% of the food-grade material. This sorting activity produced material used for further decontamination on a semi-industrial pilot plant. — *Recycleye, London, U.K.*
www.recycleye.com ■

Gerald Ondrey

A dual-shaft mixer with 1,500-gal capacity

The purpose-built FDA-1500 mixer (photo) has a maximum working capacity of 1,500 gal and features a dish-bottom, stainless-steel mix vessel with a 50-psig heating/cooling jacket. The dome-style cover includes multiple charging ports, as well as a hinged manway for major ingredient additions and cleaning. The FDA-1500 is capable of operation under vacuum up to 29.5 in. Hg. The mixer's independently driven agitators — a 40-hp two-wing anchor and a 150-hp high-speed disperser — are supported from an agitator bridge. The disperser shaft runs up to 695 rpm and is equipped with two 20-in. dia. sawtooth blades. The lower blade is stationary, while the upper blade is attached by means of an adjustable hub, enabling it to be raised or lowered to accommodate various batch volumes and ensure rapid wet-out of solid ingredients. — *Charles Ross & Son Co., Hauppauge, N.Y.*
www.mixers.com

New conveyor discharge adapter eases cleaning

This company has introduced a new discharge adapter (photo) as an option for its flexible screw conveyors. The adapter features a proprietary design with a tri-clover sanitary fitting that maintains the position and stability of the tube enclosing the rotating screw conveyor while eliminating a traditional lip seal with its potential to capture and accumulate material. The risks of cross-contamination and bacterial growth are virtually eliminated. Developed for a major snack manufacturer, the conveyor discharge head is suitable for food, nutrition, pharmaceutical and other sanitary processes. The discharge adapter is manufactured in stainless steel with a choice of sealed connections for transfer to bulk-bag fillers, hoppers, dryers and other downstream equipment. — *Automated Flexible Conveyor, Inc., Clifton, N.J.*
www.afcspiralfeeder.com

Goggles protect eyes from splash, without fogging

The Vader Goggle BKGOG-2010N (photo) is made to keep out splash and maintain vision in the toughest work environments. It is made with BK-Anti-FOG coating, a durable anti-scratch treatment, and D3-rated for droplet and splash protection. Washable and durable, BK-Anti-FOG protection lasts much longer than alternative anti-fog treatments, dispersing water molecules across the surface of the lens to restrict moisture buildup. With its wide 180-deg peripheral vision and face coverage, the Vader is also roomy enough to wear over most prescription eyeglasses. — *Brass Knuckle Safety Products, Alpharetta, Ga.*
www.brassknuckleprotection.com

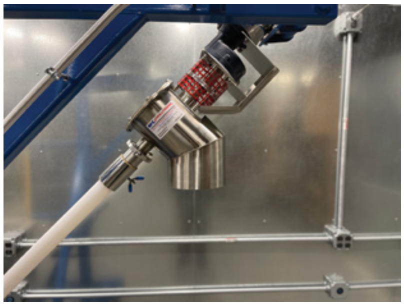
A single-use micropump for microdosing applications

The new Quattroflow QB2-Standard (QB2-SD) micropump (photo) is the smallest size in this company's family of single-use, lightweight and compact rotary microdosing pumps. The pump is designed for precision and can be used to transfer delicate biologic media. The QB2-SD has a minimum resolution of 25 µL and can handle flowrates up to 2.7 L/h, which makes the QB2-SD pump suitable for liquid-handling operations requiring precise dosing of products that can be found in cell and gene therapy, laboratory and other small-scale applications. The positive-displacement pump is lightweight (6.4 g) and compact. These single-use pumps simply click in and out of the motor drive, allowing for quick and cost-effective changeovers with minimal risk of cross-contamination. — *PSG Biotech, Oakbrook Terrace, Ill.*
www.psgdover.com/biotech

PCB terminal block for single-pair Ethernet

Single-pair Ethernet (SPE) is a powerful technology for realizing Industry 4.0 and IIoT applications. The Combicon range PCB (printed circuitboard) terminal blocks (photo) provide suitable connection technology. The networking of smart devices continues to advance. This applies in particular to sensors that are often exposed to higher-altitude ambient conditions when used in the field. In addition to the possibility of integrating an IP-protected connector into such field de-

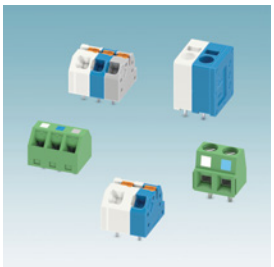
Charles Ross & Son



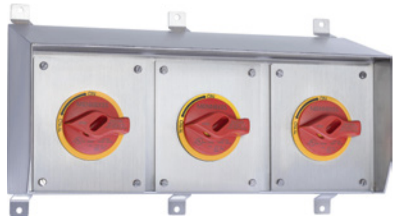
Automated Flexible Conveyor

Brass Knuckle Safety Products

PSG Biotech



Phoenix Contact



Mennekes Elektrotechnik

Denios US

Indco

Emerson

vices, there is also the option of using a cable gland to achieve the desired degree of protection. This means that the PCB terminal blocks, tested and suitable for SPE data transmission, can be used inside the devices. With clear color coding and intuitive handling, the blue and white wires of the SPE cable can be contacted clearly and safely. — *Phoenix Contact GmbH & Co. KG, Blomberg, Germany*
www.phoenixcontact.com

These disconnects have a sloped-top clean design

The new SLPX Series of sloped-top motor disconnects (photo) enable plant operators to more easily prioritize sanitation while meeting requirements for multiple disconnects. Available in three multi-gang enclosure sizes, the stainless-steel SLPX Series features a unique clean design for critical washdown and processing areas. Units are UL Listed, rated NEMA 4X and include a 15-deg sloped roof to facilitate liquid runoff. They are also suitable for multiple disconnect requirements, reducing cost and time with one-box installation. These enclosures are designed for 30- and 60-A switches and have early break auxiliary contacts for variable frequency drive (VFD) and starter interfaces. — *Mennekes Elektrotechnik GmbH & Co. KG, Kirchhundem, Germany*
www.mennekes.com

These hazardous-materials storage buildings have FM approval

This company recently received FM approval for its Rack Fire Protect (RFP) non-occupancy buildings. Fire rated for 2 hours at 2,192°F, they minimize the risk of fire spreading, making them suitable for the storage of flammable chemicals, lithium-ion batteries and other volatile materials. The adjustable racking system can accommodate up to forty-eight drums or twelve intermediate bulk containers (IBCs). Whether in single or double-tiered configurations, these designs effectively organize hazardous materials, ensuring proper segregation. Proven in hundreds of installations worldwide, they are now manufactured in the U.S. for North American installations. — *Denios US, Inc., Louisville, Ky.*
www.denios-us.com

Custom mixing systems optimize unique processing tasks

This company's custom mixing systems (photo) are engineered to optimize unique processing projects, including batch size, material properties, agitation levels and more. Engineers can develop a mixing system that includes mixers and tanks from simple open-top designs to full ASME jacketed vessels. Whether temperature is controlled to ensure viscosity of materials, to utilize heat as a catalyst, or for other reasons, jacketed tanks are often a crucial element of custom-mixing system designs. In addition to a fully integrated mixing tank and mixer design, a host of other features are available, including polished and electropolished surfaces, dip tubes and drain-valve designs and more. — *Indco, Inc., New Albany, Ind.*
www.indco.com

Liquid-level controller for oil-and-gas separators

The Fisher Fieldvue L2t liquid-level controller pairs with an electrically-actuated control valve to reliably maintain ideal separator levels. Compared to alternatives using electric on-off valves, the new Fisher controller makes it possible to save significant energy and maintenance costs by minimizing valve movement, and by simplifying the level measurement and control processes. The L2t uses a displacer to measure level and level interface, while executing a single-loop control algorithm to drive an electrically-actuated control valve. The device is simple and inexpensive to install, and there are no emissions since the valve is actuated by electricity instead of natural gas pressure. — *Emerson, St. Louis, Mo.*
www.emerson.com/fisherl2t

Safe combustible-dust explosion isolation with this system

The new and improved Exkop isolation system (photo, p. 21) is now available for many more applications, such as ST 2 dusts, reduced explosion pressures (P_{red}) of up to 2 bar and larger diameters. Also, the new Exkop Express controller allows for much greater connectivity of site process-safety systems. Process equipment is usually connected by pipelines

through which, if an explosion occurs, fire and pressure can spread very rapidly. Propagating explosions pose increased risks of injury, equipment damage and secondary explosions. The intensity of the explosion in connected vessels is increased by pressure piling and flame jet ignition. This sequence of events can be prevented by the Exkop isolation system. — *Rembe Inc., Fort Mill, S.C.*
www.rembe.us

IoT connectivity for isolated assets

The Wilsen.valve and Wilsen.node internet of things (IoT) sensor nodes (photo) wirelessly transmit data from spatially isolated assets to the network communication system. The battery-operated devices record measurement data, position data and the status of the connected sensors and wirelessly transmit this information to the IoT system via LoRaWAN, allowing monitoring and associated follow-up actions to be automated with minimal effort. Up to two inductive dual sensors for valve position feedback can be connected to the Wilsen.valve. A single device can therefore report the position of up to two 90-deg manual valves. Various types of two-wire sensors can be connected to the Wilsen.node. Typical applications for this version include object detection tasks, such as monitoring manhole lids and gates. — *Pepperl+Fuchs SE, Mannheim, Germany*
www.pepperl-fuchs.com

Convey both lean and dense phase in same system

The VS pneumatic vacuum-conveying system (photo) automatically transfers bulk materials in a choice of lean or dense phase using the same conveying system. Featuring a designed-in ball valve in the feeding elbow, the conveyor can be operated in dense phase then switched on the fly to lean or dilute phase, or vice versa, by adjusting the ratio of air to vacuum at the valve. Switching from transferring light bulk density materials, such as flour, coffee or fused silica at high velocities in suspension to transferring heavier bulk-density materials or abrasives, such as salt, flyash or mixtures at gentle, low velocities is quick, easy, and does not require tools. The hygienic vacuum conveyor includes stainless-steel construction as standard, disassembles easily for cleaning, and is ATEX-certified as explosion-proof for safe transfer of ignitable powders. — *Volkmann USA, Bristol, Pa.*
www.volkmannusa.com

Save energy in gas scrubbers with this optimizer

Launched last December, the High Efficiency Scrubber (HES) Optimizer (photo) is a digital tool that combines internal process calculations with measurements available at site to enable energy savings in the operation of Venturi-type scrubbers in wet-gas cleaning plants. Although the HES effectively captures impurity particles in wet-gas-cleaning plants, a significant pressure drop is required to achieve this. Precise adaptation of the pressure drop to the process conditions — the capacity and duty of the wet-gas cleaning in general — can enable substantial energy savings while meeting performance targets. This is exactly what the HES Optimizer is designed to do. — *Metso Corp., Espoo, Finland*
www.metso.com

High productivity chocolate forming with this machine

The Rotoform HP (high performance) is the latest model in this company's high-capacity Rotoform rotary drop depositor, which is specifically designed for chocolate processing. A pump delivers molten chocolate to the Rotoform via heated piping. The Rotoform itself consists of a heated stator, which is supplied with liquid chocolate, and a perforated rotating shell that turns concentrically around the stator to deposit drops of chocolate onto a continuously running steel belt. Baffles and internal nozzles provide uniform pressure across the whole belt width, ensuring that chips are of uniform shape and size. The heat of the drops is transferred to cooling air blown onto the product and also to the belt itself. A short cooling time means that very little oxygen can penetrate the product. — *IPCO Germany GmbH, Fellbach, Germany*
www.ipco.com

Rembe

Pepperl+Fuchs

Volkmann USA

Metso

AmorSui

This lab coat is made of recyclable materials

This company has developed a new personal protective equipment (PPE) garment for laboratory workers that is said to be 100% recyclable, while also providing enhanced safety benefits. Traditional garments worn in laboratories are made of unrecyclable materials and frequently end up in landfills at the end of their useful life, creating a great deal of waste from research and manufacturing facilities. This company's lab coat is designed to be worn over 50 times without losing fluid-repellency or anti-odor efficacy, and once it is time for a new one, the used coats can be returned to the company to be sustainably recycled. The flexible fabric features a breathable design and is also machine washable. — *AmorSui, Inc., Philadelphia, Pa.*
www.amorsui.com

3M Co.

This protective headset can charge itself using ambient light

At the CES 2024 event last month, this company unveiled what is said to be the world's first self-charging protective communications headset (photo) that converts outdoor and indoor light into clean electrical energy. The Peltor WS Alert XPV headset uses a patented solar-cell technology called Powerfoyle that recharges a built-in lithium-ion battery and eliminates the need for single-use batteries. This means that whether users are working outdoors in the sun or inside with artificial light, their headset will continuously charge. In addition to light-harvesting capabilities, the headset includes many advanced features that allows workers to stay safe, connected and productive. It can connect with Bluetooth technology-enabled devices and offers a noise-cancelling microphone to enable easier and clearer conversations, even in noisy work environments. The push-to-listen feature allows users to hear their surroundings without removing the headset by pausing Bluetooth streaming and activating the ambient microphones to listen to nearby colleagues and sounds. Users can quickly adjust the settings using the headset's glove-friendly push buttons. — *3M Co., St. Paul, Minn.*
www.3m.com

Xchanger

Multi-stage condenser for cracking organic waste material

The multi-stage TV-200 condenser (photo) was designed to separate bio-waste materials produced during an anaerobic process known as pyrolysis, which applies heat to break down organic waste into biopolymer components. Each stage of the TV-200 cools the deconstructed biopolymers, which condense at varying temperatures, separating tars, phenols and organic acids. With the TV-200 condenser, what was waste material can now be recycled as biofuel to supplement power, carbon products used in fertilizer or filter media and acetic acids used in commercial products. Because oxygen is not present in pyrolysis, no additional CO_2 is generated, and carbon products are not released back into the atmosphere. The assembly includes a custom mounting frame and instrument ports to monitor the performance at each stage. — *Xchanger, Hopkins, Minn.*
www.xchanger.com

Siemens

New partnerships bolster spatial simulation and regenerative AI

This company's Xcelerator portfolio of industrial software is being combined with Sony's new spatial content-creation system, featuring the XR head-mounted display with high-quality 4K OLED microdisplays and controllers for intuitive interaction with three-dimensional objects (photo). Enabling designers and engineers to create and explore design concepts in a borderless immersive workspace, the new platform integration will kickstart content creation for the industrial metaverse. First announced in January at CES 2024, and expected to be available later in 2024, NX Immersive Designer, an integrated solution that combines the NX software platform and Sony technology, brings immersive design and collaborative product engineering capabilities to this company's flagship product engineering solution. At CES, this company also announced an expansion of its partnership with Amazon Web Services (AWS) to build and scale generative artificial intelligence (AI) for industrial applications. — *Siemens AG, Munich, Germany*
www.siemens.com ■

Gerald Ondrey and Mary Page Bailey

Overview of Reactor Types

Department Editor: Scott Jenkins

Reactors are at the core of most processes in the chemical process industries (CPI) and are broken down most broadly by whether their operating mode is non-flow (batch) or continuous. Each reactor type has advantages and disadvantages depending on the phases and properties of the reactants, the thermodynamics and kinetics of the reactions and the type of products being generated. This one-page reference provides a refresher on the most common types of reactors used in chemical manufacturing.

Batch reactors

Batch reactors are closed vessels where reactants are added sequentially. Stirred tanks, with an agitator to mix the reactants thoroughly, are the most common batch reactor. Stirring mixes the ingredients initially, maintains homogeneity during the reaction and enhances heat transfer at internal surfaces and jacket walls. Batch processing is used generally when the reaction times are long or the required production volumes are small.

Semi-batch reactors are modified versions where reactants are periodically added or products are periodically removed. Semi-batch reactors may offer greater control over yield or selectivity of the products. This reactor type is useful for carrying out exothermic reactions because the flow of added reactants can be varied to better control the reaction. Scaling up semi-batch processes generally has higher capital costs than those for continuous process reactors.

Continuous stirred-tank reactor

In a continuous stirred-tank reactor (CSTR), reactants are continuously fed into the reactor vessel, where an agitator mixes them to produce desired products, which are removed continuously from the reactor.

The agitator maintains a constant concentration throughout the reactor. Mixing times (length of time needed to achieve homogeneity of mixture of inputs) depends on the geometry of the vessel and the speed and power of the agitator. The average residence time for fluid inside the tank at steady-state flow is the ratio of the total reactor volume to the volumetric steady-state flowrate of fluid exiting the reactor.

One major advantage of employing a CSTR is generating a massive product volume. These are continuous reactors that can be run for extended periods. CSTRs are unsuitable for reactions with extremely slow kinetics.

In many cases, CSTR reaction proceses are carried out in a series of reactors, called CSTR cascades, to provide higher conversions.

Plug flow reactors

In plug flow reactors, also known as tubular reactors, reactants and products flow through a cylindrical pipe with openings at each end. "Plugs" of reactants are continuously fed into the reactor, and as the plug flows along the length of the reactor, the reaction takes place. This results in an axial concentration gradient. Products, along with unreacted reactants, continuously exit the reactor.

Plug flow reactors are mechanically simple and easy to maintain, with high conversion rates for a given reactor volume generally being observed. Because reactor temperature is difficult to control, these reactors can be suboptimal for exothermic reactions.

Fixed-bed reactor

Often deployed for catalytic processes, fixed-bed reactors are versatile, used in applications such as absorption, distillation, stripping, separation processes and catalytic reactions. Although the physical dimensions of the beds can vary greatly, typical fixed-bed reactors consist of a cylindrical chamber containing catalyst particles or pellets (Figure 1). Fluid flows through the catalyst bed, allowing the desired reaction to occur. Fixed beds contain catalyst particles that usually fall into the range of 2–5 mm dia. Catalysts can be loaded in several ways, including a single large bed, several horizontal beds, several packed tubes in a single shell, single bed with embedded tubes and beds in separate shells.

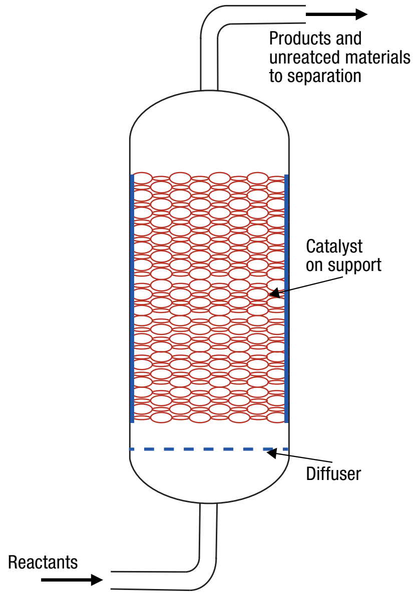


FIGURE 1. Fixed-bed reactors, like the one shown in the diagram, are versatile, used for catalytic processes, separations and others

Fluidized-bed reactor

In fluidized-bed reactors, a heterogeneous catalyst is fluidized by an upward-flowing gas or liquid This allows for extensive mixing in all directions. A result of the mixing is excellent temperature stability and increased mass transfer and reaction rates.

Fluidized-bed reactors must be designed so that the fluid flowrate is sufficient to suspend the catalyst particles. The particles typically range in size from 10–300 µm.

When designing a fluidized-bed reactor, the catalyst life must also be taken into account. Most fluidized-bed reactors have a separate system to regenerate the catalyst. ■

References:

1. Couper, J.R., Penney, W.R., Fair, J.R. and Walas, S.M., "Chemical Process Equipment: Selection and Design, 2nd ed.," Gulf Professional Publishing, Burlington, Mass., 2005.
2. EPCM Holdings, Chemical Reactors: Types and Operations, Pretoria, S.A., epcmholdings.com, accessed December 2023.
3. Essential Chemical Industry, Chemical Reactors, essentialchemistryindustry.org, March 2013.
4. Dhodapkar, Shrikant V., Klinzing, George E. and Zaltash, A., A Primer on Gas-Solids Fluidization, *Chem. Eng.*. August 2012, pp. 38–47.

Innovations in Pilot-Plant Distillation Process Design

Small-scale distillation processes have specific concerns that are different than commercial-scale distillation. Presented here are several topics, including column internals and column-sizing software, considered from the perspective of pilot-scale distillation

Glenn Graham and Raymond Rooks
AVN Corp.

IN BRIEF

COLUMN INTERNALS

FLEXIBLE DISTILLATION COLUMN

COLUMN-SIZING SOFTWARE

DECANTERS

POLYMER FORMATION

The design and operation of laboratory- and pilot-scale columns are greatly influenced by their purpose, the effect of scale on engineering principles, the availability of equipment, and the short-term nature of projects. Some of the main considerations are listed here, and these often require innovative solutions to meet the project requirements:

- There are significant differences in the type of equipment available, such as pumps, flowmeters, and control values, the types of trays and packing used, and the facilities required. The facilities can also affect the column design, especially regarding column height
- For separations problems in which data are the main product, small-scale equipment must have appropriate sample points or on-line analyses capabilities, and instrumentation that may not be necessary for an equivalent commercial column, and often must be able to operate at a wider range of conditions than for a commercial column
- For each separation, there are always many options, and it is usually a compromise based on equipment size, type, and features, which are influenced by cost, time, and equipment and facilities availability
- The short-term nature of most laboratory- and pilot-scale projects emphasize the importance of equipment versatility and ease of assembly. Flexible systems that can be easily modified are very desirable

In a previously published article (*Chem. Eng.*, March 2023, pp. 23–30), we highlighted the specific concerns imposed on the purpose, design, and operation of small-scale distillation equipment and discussed how these concerns manifest themselves in the actual equipment that is typically used. The topics included the challenges of scale, high-vacuum operation, heat control, reflux control, distillate and sidestream product takeoffs, and reboilers.

This article continues the discussion by covering additional aspects that should be considered when dealing with small-scale distillation equipment. The following topics are discussed:

- Column internals
- Flexible distillation column concept
- Column-sizing software
- Decanters
- Free-radical polymer formation

Column internals

Column internals, whether trays, structured packing or random packing, play a large role in distillation effectiveness. In pilot-scale processes, there are a number of design considerations relative to column internals, and some key differences compared to large-scale distillation.

Trays. Glass Oldershaw-style trays [*1*] are commonly used for laboratory- and small pilot-scale distillation columns. Figure 1 shows

FIGURE 1. Glass Oldershaw trays are available "off the shelf"

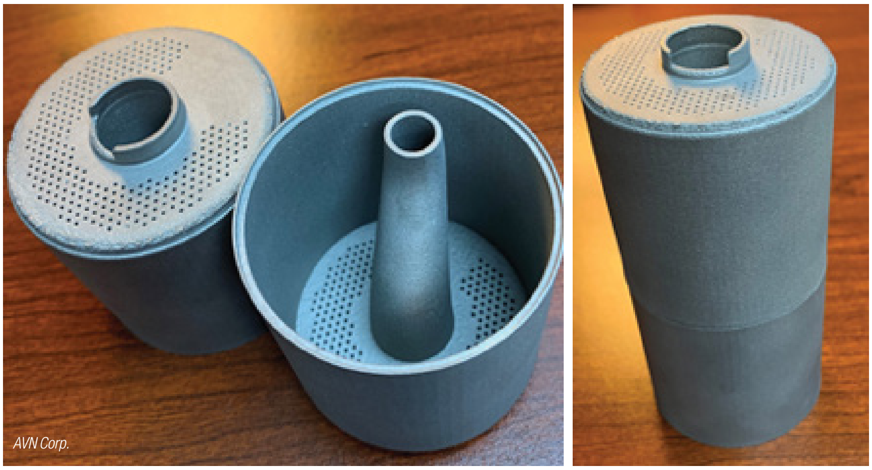


FIGURE 2. Oldershaw trays can also be made from 3D-printed stainless steel, as well as a variety of other metals

FIGURE 3. Ceramic saddles (left; photo used by permission from Chemglass Life Sciences) and metal Pro-Pak packing (right; photo used by permission from Performance Distillation Solutions) are examples of random packing

an example of an unjacketed section. They are available as "off-the-shelf" components in a range of diameters from 28 to 100 mm, with up to 30 trays per section (usually in five-tray increments), in vacuum-jacketed or bare column sections. The trays available from each vendor have dimensions for the number of holes, hole diameter, and downcomers that are fairly similar. Custom variations of the column sections for themowells, feed or sample ports and connection joints are common. Custom designs for the tray dimensions are possible, but tend to have high setup costs.

Due to the high degree of mixing for liquid on the trays and for the vapor, the efficiency for Oldershaw trays is a point efficiency [*2*]. For commercial trays, in which the liquid on the entire tray is not as well mixed, the composition can vary significantly as the liquid moves across the tray, which increases the overall tray efficiency above the point efficiency. The extreme case for this is a Lewis Type 2 efficiency, where the vapor is not mixed and the liquid flow is entirely in one direction. Large-column efficiencies can exceed 100%, depending on the definition of efficiency [*3*]. Therefore, if a specific separation can be achieved in the laboratory with a certain number of Oldershaw trays, the separation in a commercial column would normally be expected to be at least as good with the same number of trays. This makes it relatively easy to scale up Oldershaw column data. One caveat is that this conclusion is not valid if cellular foam is present. Trays also have an advantage with high-purity service, since they ensure good mixing and their design prohibits channeling of gas and liquid, in which a small amount can lead to product impurities.

One of the other key advantages of using glass trays is the ability to observe the tray activity and determine if there are issues related to variation in tray activity between trays, foaming, fouling and color formation. The pressure drop is usually in the range of 1 to 2 mm Hg per tray. This is an advantage for laboratory columns with many trays or those that are operating under high vacuum, since the reboiler temperature will be not be excessively elevated due to high pressure drop in the column. However, care must be taken when scaling up these data, since commercial column trays will usually have significantly higher pressure drops. This means that not only will the reboiler temperature be higher for the same head pressure as the laboratory column, but the separation may actually be affected if there is a significant effect of pressure on the vapor-liquid equilibrium, which is especially true at vacuum conditions.

One of the disadvantages of using glass is the pressure constraint, which limits operation to atmospheric pressure (or very slightly above) and below. Metal Oldershaw column sections are not commercially available. It is possible to custom build these, but this option is difficult and expensive by traditional methods. However, work by the authors' company, along with the Robert C. Byrd Institute, suggests that metal trays can be fabricated using additive manufacturing (3-D printing). Examples are shown in Figure 2. Each tray and its surrounding column are printed with appropriate grooves that easily allow several tray units to be welded together to create sections with any desired number of trays, as well as several different materials of construction, such as stainless steel, high-nickel alloys and titanium. Thermowells, sample ports and feed inputs can easily be added.

High precision is possible for the creation of these trays, including the hole dimensions and locations. They are relatively inexpensive to make and design customization is not difficult, which is an additional advantage over glass trays.

Random packing. This type of vapor-liquid contactor is often used for small-scale distillation columns as an alternative to trays. This consists of

FIGURE 4. Structured packing has a low pressure drop (photo courtesy of Sulzer Chemtech)

an open tube with a packing support, into which the packing can be poured. The HETP (height equivalent to a theoretical plate) is very low for most laboratory random packings, allowing for a relatively large number of stages in a location with limited height, such as under a typical laboratory fume hood. Pressure drop per stage is generally lower than for trays, making random packing especially good for vacuum operation. If fouling occurs in the packing, the packing can be easily replaced.

However, scaleup of distillation setups using random packing is more difficult than for Oldershaw trays. Liquid distribution and collection are important considerations, as well as the surface area of the column wall. For Oldershaw trays, the liquid from the tray above, or from a liquid feed stream, can simply be fed onto the tray where it will be mixed by the bubbling action on the tray. Packing requires that the liquid be properly distributed onto the packing. For columns up to about 3 in. in diameter, only a single tube located in the center of the column and just above the packing is sufficient for good liquid distribution (multiple points are required for larger-diameter columns). Between 2 and 3 in. in diameter, some additional packing may be required due to suboptimal distribution. This is not difficult for the introduction of reflux at the top of the column, but for feed sections, the liquid from the packed section above must be collected, mixed with the feed, then distributed to the packing section below.

It is also important that the random packing elements are not too large relative to the column diameter. The general rule-of-thumb is that the column diameter should be at least eight times the approximate diameter of the packing. However, it is important to check with packing suppliers for their recommendations.

Figure 3 shows examples of two common types of random packing. Figure 3 (left) shows ceramic saddles. These are very inexpensive and are resistant to acidic corrosion. However, they have relatively high pressure and high HETP. In addition, the smallest ceramic saddles are around 0.25 in. in diameter, which means that they should not be used in columns less than 2 in. in diameter.

Figure 3 (right) also shows an example of Pro-Pak [*4*] (Performance Distillation Solutions; spun off from Cannon Instrument Co.) packing. This packing has low pressure drop and low HETP and comes in two sizes (0.24 and 0.16 in.). It can be obtained in a variety of metals, including Hastelloy C. Distillation data are available to assist with column design. This type of packing is relatively expensive, but the cost is partially offset by the large number of stages per volume of packing purchased, for a given column diameter. At low vacuum, there is some uncertainty in efficiency, so HETP testing should be performed for a specific service.

Structured packing. Figure 4 shows an example of structured packing, which is available for laboratory- and pilot-scale columns from Koch-Glitch [*5*] and Sulzer [*6*], such as DX and EX. This type of packing has the lowest pressure drop per theoretical stage of any vapor-liquid contactor discussed so far. Its most important application is in high-vacuum service, where minimizing pressure drop is important. The HETP can be relatively low also. Sulzer does not have scale up information for DX/EX gauze packing, only for CY and BX. Testing with model systems at the same pressure and flow rates can be used to compare the packing efficiency of DX/EX with commercial packing. At elevated pressure, the HETP for gauze packing can appreciably increase (more height required) as the liquid layer thickness builds on the gauze, eliminating the advantage of gauze over sheet metal.

FIGURE 5. Vigreux columns, such as the one shown here, are used for crude separations

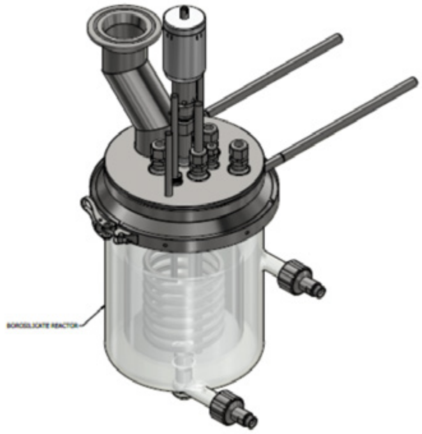


FIGURE 6. Flexible reboilers, like the one shown here, have a heating coil, jacket and metal headplate (drawing used with permission from H.S. Martin, part of the AGI Group)

Structured packing has similar liquid distribution and collection requirements to those of random packing. Structured packing generally has long lead times (6 months) since it has to be custom-made for a specific column size. There are significant pricing variations depending on vendor/materials.

Vigreux column. This type of column, an example of which is shown in Figure 5, is often used by chemists to make crude separations. It has very low pressure drop and low liquid holdup, and is very inexpensive. For a 1-in. diameter column, the HETP is in the range of 4 to 6 in. [*7*]. Vigreux columns work well for small-scale separations where low pressure drop is needed (high-vacuum operation), a low number of stages is required, visual observation is desirable (Vigreux columns are constructed of glass), and there is a strong desire to minimize expenses.

Flexible distillation column

Flexible distillation columns refer to those designed to be reconfigurable for multiple options. This type of laboratory-scale column has reconfigurable reboilers and column sections.

Reboiler. Figure 6 shows a diagram of a flexible reboiler to be used as

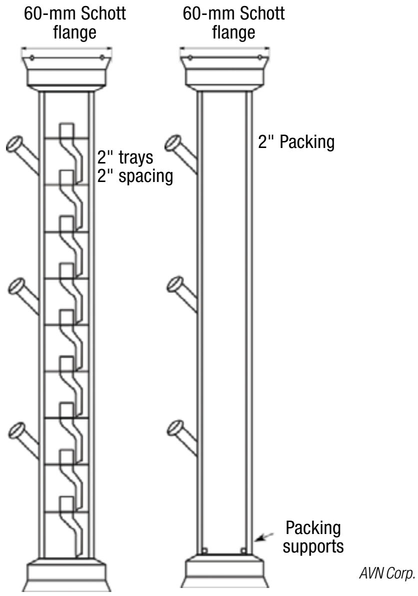


FIGURE 7. **Flexible column sections can contain trays or packing**

FIGURE 8. **The photo shows a joint heater for 60-mm Schott flange on a 1-in.-dia. packed and unsilvered distillation column**

part of a versatile laboratory distillation apparatus. The glass vessel can be heated by hot oil in its jacket or its coil. A bottom port allows connection to a forced-circulation system. The glass vessel can be replaced by an unjacketed version that would allow for heat input by an electric heating mantle.

The hollow headplate, which is constructed of Hastelloy C, can be heated with hot oil to prevent vapor condensation on the headplate. A magnetic-drive agitator is mounted in the center of the headplate, which also has connections for the column, a thermocouple, liquid level and column differential pressure (high lead), forced circulation return and sample collection. Agitation becomes critical at low pressure, due to liquid bumping (vapor-liquid disengagement).

Seals between the headplate and column, as well as the headplate and glass vessel, are made of FFKM perfluoroelastomeric O-rings (Kalrez, Simrez, Markez and so on), which are resistant to a large range of chemicals. Some formulations can withstand temperatures in excess of 300°C, and provide nearly leak-free connections even at high vacuum. At elevated pressures, however, other joint types should be explored.

A reboiler such as this would be intended as the centerpiece of a versatile distillation system that will allow for rapid exchange of packed or trayed sections, as well as modifications to the number of stages, number and location of feeds, condenser types and inclusion (or not) of a vacuum system, a sidestream or an overhead decanter. The heating bath has the capability of heating oil up to 300°C.

Column sections. Figure 7 shows examples of column sections to be used in this distillation system. These contain Oldershaw trays, or are open tubes used for random or structured packing. O-ring joints (60-mm Schott Duran O-ring joints) allow connection to the metal headplate discussed above, or to each other. Multiple O-ring ball-joint column ports can be used for feed, sampling or thermocouples. These types of joints have the advantage (over ground-glass joints) of low clearances, so that the entire column does not need to be disassembled to make a change to a column section. They do not require vacuum grease, which can lead to process contamination. The joints don't freeze together, which would prevent disassembly, and they provide more consistent vacuum sealing performance. In addition, the clamps are easy to install. In order to prevent heat loss from the joints, custom joint heaters have been fabricated (Figure 8).

Normally, adiabatic operation will be accomplished by heat tape and insulation. However, vacuum-jacketed column sections can also be used.

The 3D-printed tray sections, as discussed earlier, can be used in place of the glass sections for pressure operations, assuming that the other glass parts on the system will be replaced with metal. Alternative joint types will need to be used, such as sanitary seals, pipe, Grayloc and so on.

Column sizing software

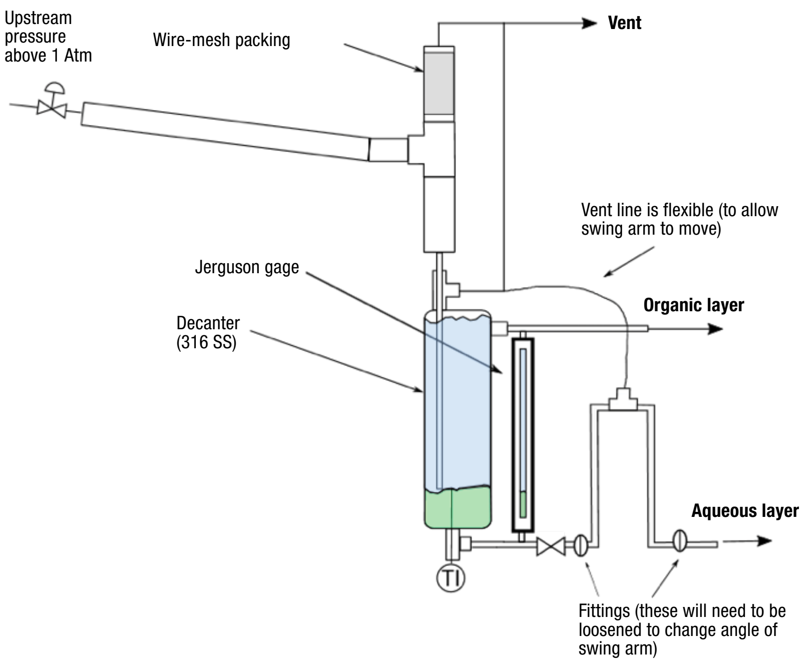


FIGURE 9. **Automatic decanters can be used for distillates with two liquid phases**

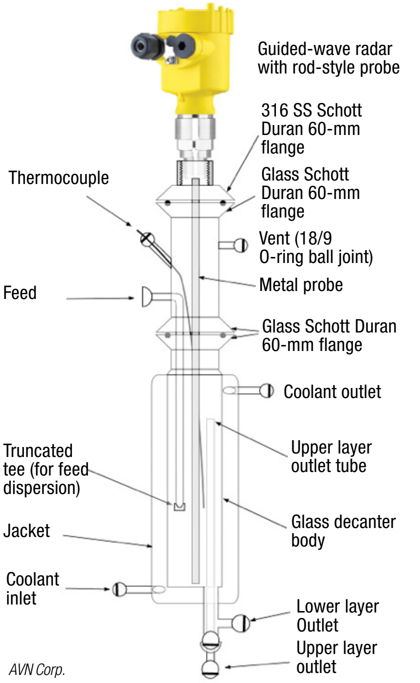


FIGURE 10. This diagram shows an automatic decanter that is controlled based on signals from a guided-wave radar instrument

Process-simulation software, such as Aspen Plus, is capable of determining the size requirement of trayed or packed commercial-scale columns. However, the type of trays and packing typically used in small-scale columns are not included in the column internals calculation blocks of the process simulation software.

Proprietary software for sizing laboratory- and pilot-scale trayed and packed distillation columns is available. This software allows for rapid comparisons between trayed and packed column to assist in the most appropriate type and size for column specifications. The programs are being converted to web-based tools. Other tools for small-scale distillation, such as for reboiler and condenser design, are anticipated in the future.

Decanters

For chemical systems with heterogeneous azeotropes, in which the distillate consists of two liquid phases, a decanter will usually be required. Two of the main concerns are (1) the residence time required for phase separation and (2) how the removal of each phase will be controlled. Determination of the residence time will not be covered here.

Depending on the ratio of the phases, the flowrates and the degree of sophistication of the equipment setup, decantation can sometimes be controlled manually. However, it is usually desirable for this operation to be automated.

The most simplistic means of automating a decanter is through the use of hydrostatic pressure [*8*]. Figure 9 shows an example of a pilot-scale decanter based on this principle. A dip tube enters the top of the decanter and extends to the approximate level of the liquid-liquid interface (the diptube generally has a momentum breaker). The light phase (usually organic) simply overflows from a port toward the top of the decanter, low enough to leave a vapor space above the liquid for venting. The heavy phase flows out the bottom of the decanter and into the bottom of an inverted U-tube, which is vented at the top of the U-tube. The U-tube has two joints that allow it to be rotated to adjust the height of the top of the U-tube relative to the decanter overflow drain. The hydrostatic head in the decanter, which consists of the light and heavy phase, must equal the head in the U-tube, which contains only the heavy phase. The height of the interface will automatically adjust itself until the hydrostatic pressures are equal. The interface would normally be maintained at a level in which the mass ratio of the two phases is equal to their ratio entering the decanter, so that the residence time of each phase is about the same.

For the metal decanter depicted in Figure 9, a Jerguson gage is connected between the liquid overflow drain and the decanter drain to allow visual observation of the interface level inside of the decanter. In this design, the feed to the atmospheric decanter was vented from a higher pressure through a depressurization system to eliminate disturbances of the interface in the decanter.

Glass versions of this decanter, based on the same hydrostatic concepts, are routinely used for glass laboratory- and small-pilot-scale columns. For atmospheric columns, smooth operation of the decanter is usually easy to achieve. However, if the column operates under vacuum, or at elevated pressure (in a metal column), care must be taken to ensure that the decanter pressure is controlled well. Fluctuations in the pressure can cause a false pressure difference between the decanter and the U-tube, causing the interface to move. In these situations, as an alternative to the hydrostatic head principle, a capacitance probe or guided-wave radar, tied to a control valve or pump below the decanter bottom drain, can be used to control the interface level.

Figure 10 shows another style of decanter that uses the input from a guided-wave radar instrument to maintain the overall and liquid-liquid interface levels by controlling the flowrates of the upper and lower liquid layers. This type of instrument is able to detect both the overall and liquid-liquid interface levels because, as the radar signal is transmitted down the metal probe, part of the signal bounces from the top surface, but the remainder of the signal continues down to the liquid-liquid interface, where it is reflected back. The rod can be cut with a metal saw and can be replaced to change the length or materials of construction (possibly to an exotic metal, such as Hastelloy).

The body of the decanter is glass (although it can be easily swapped

FIGURE 11. Friedrich condensers may be necessary to insulate the condenser to avoid condensation on the outer wall (photo and drawing used with permission from Chemglass Life Sciences)

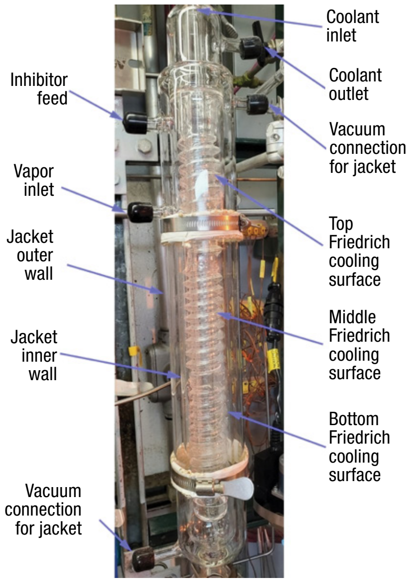


FIGURE 12. The condenser shown here has three Friedrich sections. In addition to increased surface area, it also has a jacket that can be evacuated for insulation and enhancements for inhibitor introduction. This jacket prevents condensation on the wall, which may not have an inhibitor (Photo used with permission from H.S. Martin, part of the AGI Group)

out for an all-metal design), which allows for visual observation of both liquid layers. The decanter also has a jacket for coolant. The connection joints are O-ring ball joints, which allow for very good sealing without the use of grease. A small diameter (1/16-in.) thermocouple can be inserted down into the liquid. Feed material flows into the center of the decanter through a truncated tee that directs motion horizontally to minimize disturbances of the liquid-liquid interface.

The upper layer leaves the decanter through the upper-layer outlet tube. The lower layer flows out the bottom of the decanter through the annualar space around the upper-layer outlet tube. The overall level is maintained slightly above the level of the coolant outlet port. The height difference between the overall liquid level and the top of the upper layer outlet tube provides room for process control of the overall level. By controlling the outlet flowrate of the lower layer, the interface level can be maintained, whereas controlling the outlet flowrate of the upper layer maintains the overall liquid level.

Due to the two 60-mm Schott Duran flanges, the design of the decanter is very modular. If the diameter or height of the decanter needs to be changed, the decanter body can be replaced. The upper-layer outlet tube can be easily replaced with a different length. The feed tube length can be modified by a glass blower by removing the upper glass piece. Glass will accommodate acidic liquids and, as mentioned earlier, the guided-wave radar rod can be replaced with an exotic metal.

A consideration that affects how the decanter is connected to the column is that of which liquid phase is refluxed to the column. If both liquid phases are refluxed in equal proportions, it may be possible to use a liquid dividing head to split the condensate for reflux and send the distillate to a decanter, although this can be tricky if there is strong phase separation between the liquids. If only one liquid phase will be refluxed, then the condensate should be decanted first and the refluxed layer can then be sent to the reflux splitter. For the cases in which both phases need to be refluxed, but at different ratios, the condensate should first be decanted, then each phase should refluxed separately (separate reflux splitter or pumps).

Polymer formation

Polymer formation is a special problem for distillation columns. Some examples of chemicals that easily polymerize are acrylates, acrolein, acrylonitrile, acrylic acid and styrene. The difficulties posed by these compounds are associated with how easily they polymerize, the solubility of the polymer in the monomer, and how difficult it is to inhibit the polymerization.

Examples of polymerization inhibitors are phenothiazine, hydroxyquinone, quinone, and 4-hydroxytempo. Oxygen itself hinders polymerization, but it also activates some inhibitors. Metal surfaces, such as copper and Monel alloys, also hinder polymerization.

The inhibitors listed above have low vapor pressures and essentially do not exist in the vapor phase in a distillation column, so vapors condensing on uninhibited surfaces are prone to polymerization. The two main areas of a distillation column that require special attention, due to this process, are the condenser and the column.

Condenser. It is important that the cooling surface of the condenser is coated with inhibited liquid. Two of the ways to accomplish this are with the use of a Friedrich condenser for laboratory columns and a spray condenser for pilot columns.

Figure 11 shows an example of a typical Friedrich downdraft condenser. The cooling surface is a spiral inner tube with close tolerances between the spirals and the outer tube. Inhibitor solution introduced at the top tends to spiral down and coat this cooling surface. These condensers works well for small columns, but lack enough cooling surface and vapor-flow capacity for larger columns. The advantage is that this solution is simple.

An enhanced Friedrich-style downdraft condenser is shown in Figure 12. This condenser is constructed of three spiral Friedrich sections fused together for increased surface area. Inhibitor is fed to the top of the upper Friedrich section and the vapor inlet is halfway down this section. This allows the inhibitor to coat the surfaces above the vapor inlet to minimize the possibility of having uninhibited condensation surfaces, even if some of the vapor flows upward and condenses. In addition, the condenser has a jacket which can be evacuated to full vacuum or heated. This minimizes or eliminates any condensation on the outer process wall, in case this surface is not well-coated with inhibitor.

Figure 13 shows an example of a spray condenser. The condensate is pumped around in a loop, through a heat exchanger, for heat removal, and a spray nozzle located inside of a collection vessel. Inhibitor is introduced into the recirculation loop. The vapor from the column is condensed by the spray, which also ensures that all of the surfaces are coated with inhibitor.

Column. Trays work well for polymerizable chemicals because the inhibited liquid spray tends to coat most of the column walls and other surfaces. Packing lacks this spray feature and has more of a tendency to have uninhibited surfaces on the

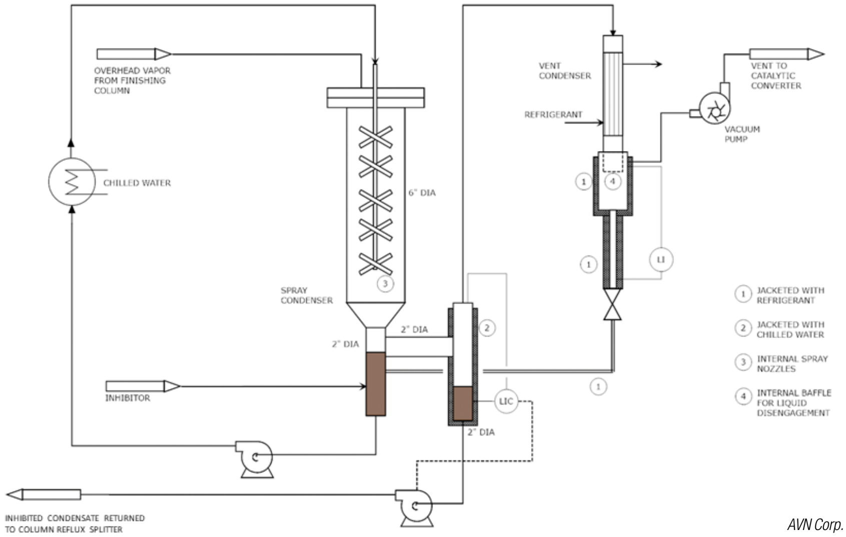


FIGURE 13. **The diagram shows a spray condenser system**

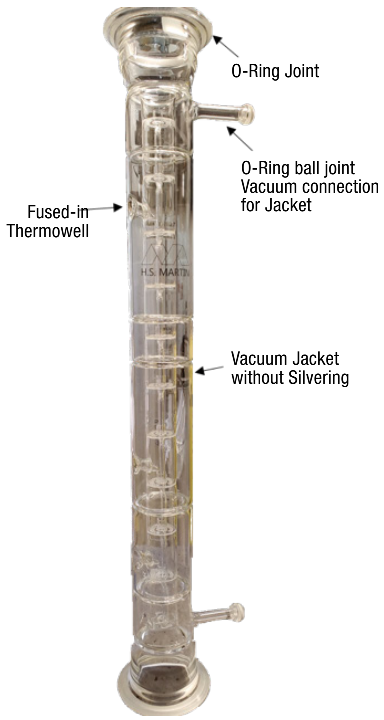


FIGURE 14. **This Oldershaw column section is designed for polymerizable chemicals. It has an unsilvered jacket that can be evacuated with a vacuum pump, fused-in thermowells, and O-ring joints that have heated mantles to prevent heat loss (photo used with permission from H.S. Martin, part of the AGI Group)**

column wall and within the packing. Packing can be used in certain cases — for example, if the polymer has at least some solubility in the monomer, but in general, has more tendency to foul due to polymer formation.

Figure 14 shows a custom Oldershaw column section designed specifically for polymerizable chemicals. It has Schott Duran O-ring joints that seal well and allow the column section to be removed with minimal vertical movement of the adjacent sections. Special heating mantles are wrapped around the joints to eliminate heat loss, condensation and potential surface polymerization.

Although most vacuum-jacketed glass Oldershaw column sections are silvered to prevent radiant heat loss, the column section show in Figure 14 was designed for temperatures that do not exceed 80°C, at which radiant heat losses are minimal. The jacket can be connected to a vacuum pump to eliminate heat loss by convection. With this arrangement, if polymerization does occur on the trays, the column section can be easily removed and placed into an annealing oven to be baked out. This treatment on a traditional silvered column section would destroy the silvering and vacuum. In this case, the column would need to have the vacuum broken, baked out with silvering removed, re-silvered, and have vacuum resealing, which is significantly more time consuming and expensive.

Traditionally, tray temperature measurements involve either thermocouples inserted directly onto the tray or removable thermowells installed just above the tray. In either case, ball joint connections are usually used for the thermowells. However, this arrangement tends to have cold spots in which condensation can occur. If the chemicals are polymerizable, the condensation may occur on uninhibited surfaces, leading to polymerization, possibly gluing the thermowell or thermocouple to the column section permanently. The column section in Figure 14 has fused-in thermowells that eliminate this issue. Fused-in thermowells can also be used for high-vacuum service (<50 mm Hg), where we wish to minimize air leaks into the system. It is important when using fused-in thermowells that the thermowell is filled with oil or a thermal paste for good heat transfer. ■

Edited by Scott Jenkins

References

1. Oldershaw, C. F., Perforated Plate Columns for Analytical Batch Distillations. *Industrial & Engineering Chemistry Analytical Edition*, pp. 265–268, 1941.
2. Kister, H. Z., "Distillation Design," McGraw-Hill, New York, N.Y. 1992.
3. Lewis, W. K., Rectification of Binary Mixtures. *Ind. Eng. Chem.*, pp. 399–402, 1936.
4. Performance Distillation Solutions; spun off from Cannon Instrument Co. (n.d.). Pro-Pak Protruded Metal Distillation Packing Literature. State College, Pa.
5. Koch-Glitsch. (n.d.). Structured Packing. Retrieved from https://koch-glitsch.com/technical-documents/brochures/structured-packing-brochure.
6. Sulzer. (n.d.). Laboratory Packings. Retrieved from https://www.sulzer.com/en/shared/products/laboratory-packings
7. Krell, E., "Handbook of Laboratory Distillation," Elsevier, Amsterdam, the Netherlands, 1982.
8. Frank, T. C., Liquid-Liquid Extraction and Other Liquid-Liquid Operations and Equipment. In D. W. Green (Ed.), "Perry's Chemical Engineer's Handbook," 9th ed., pp. 15–87, McGraw-Hill Education, New York, N.Y., 2019.

Authors

Glenn Graham is a corporate fellow at AVN Corp. (which recently acquired Matric; 1740 Union Carbide Drive, South Charleston, WV 25303; Email: glenn.graham@AVNcorp.com; Phone: 304-552-6554). Graham is responsible for providing technical guidance and support for laboratory- and pilot-scale research and development projects, including ASPEN computer simulations, the design and operation of laboratory- and pilot-scale equipment, and experimental data analyses. He specializes in distillation and related technologies. He joined AVN in 2013. Prior to joining AVN, Graham worked for Union Carbide and Dow Chemical in their R&D separations groups, also specializing in distillation and related technologies. Graham holds B.S.Ch.E and M.S.Ch.E degrees from Montana State University.

Raymond Rooks is a principal engineer at AVN Corp. (formerly Matric; same address as above; Email: raymond.rooks@AVNcorp.com; Phone: 304-720-1037). Rooks has more than 20 years of experience in the chemical industry. He has held positions at Simulation Sciences, Union Carbide/Dow, and Praxair/Linde. He has broad experience in process development, including process modeling, simulation, experimental design, and experimental/laboratory simulation, especially in process separations. Rooks holds a Ph.D. in chemical engineering from the University of Massachusetts at Amherst and a B.S.Ch.E. from the University of Oklahoma.

Heat Pumps: Decarbonizing the Process Industries

As the chemical process industries push to decarbonize their businesses, heat pumps are emerging as a go-to alternative to fossil fuels. Efficient and cost-effective, heat pumps are a rapidly expanding solution for industrial heat demand

Jörg Freckmann
MAN Energy Solutions

In the drive to meet global sustainability targets, there remains a major challenge in decarbonizing many process industries, which are responsible for around a fifth of all greenhouse gas emissions. Electrification, while possible, can present substantial efficiency challenges, which leads to the broad belief that many industrial sectors will be overly difficult to decarbonize.

In considering an alternative to electrification, the latest generation of high-temperature heat pumps offers an attractive approach. Large-scale heat pumps present an efficient mechanism to convert so-called "waste" heat or even ambient heat sources, such as the atmosphere or bodies of water, into valuable high-quality process heat.

Many facilities in the chemical process industries (CPI) rely on steam, the production of which requires a great deal of heat. Overall, steam production represents about two thirds of energy demand for the process industry sector. However, modern heat pumps are fully capable of delivering steam at temperatures up to 300ºC and at industrial volumes and pressures, even when using low-temperature heat sources. This article introduces industrial heat pump use and discusses the decarbonization potential for users of heat pumps.

The heart of the heat pump

Compression. At the heart of the latest generation of large-scale heat pumps is an efficient, robust and reliable compressor. Various kinds of compressors may be deployed for industrial heat pump applications, including scroll, reciprocating, screw and both inline and radially geared centrifugal types. In large industrial applications exceeding 10 MW_{th}, centrifugal compressors are invariably used. As an example, integrally geared compressors can be deployed for process steam applications (Figure 1). Some types of these centrifugal compressors feature a bull gear able to drive pinion shafts and attached impellers for efficient compression. Designed to meet stringent industrial standards, radially geared centrifugal compressors have a strong track record and are commonly used elsewhere in the chemical and petrochemical industries.

Refrigerants. Various refrigerants can be employed for heat pump cycles, and the thermodynamic properties of the working fluid can significantly influence the heat pump process. Making the correct choice of refrigerant is a key consideration in optimization of the thermodynamic process. Qualities such as critical temperature and pressure and evaporation/condensation enthalpy are indeed critical. But other characteristics, such as compatibility with component materials like metals or seals, and chemical stability, as well as safety and environmental factors are also important, as are costs. Commonly used refrigerants fall into three broad groups — natural, hydrocarbons and synthetic.

The performance of a heat pump with a particular refrigerant is measured as the coefficient of performance (COP), which indicates that refrigerants like ammonia, butane and propane are among the most efficient naturally occurring refrigerants. COP is calculated as the ratio between the rejected heat and compressor work. Ammonia is generally considered to have the best COP for steam production, with the highest power density. Future research focusing on heat pump refrigerants, such as azeotropic mixtures, is needed to explore potential advances aimed at further optimization of process steam generation.

Meeting industrial demands

In comparison with district heating, processes found in the CPI typically require higher temperatures and pressures. Typically, these processes are met by conventional electrical or fossil-fuel-powered steam boilers. However, the flexibility afforded by heat pumps makes them attractive to production processes in those kinds of industries. As such, conventional boilers can be replaced by industrial heat pumps, especially as one of their main advantages is the ability to use waste heat and ambient energy to deliver more heat output than the electrical energy consumed.

During a conventional heat pump cycle used for industrial process-steam production, an evaporator is located at a heat source, which is fol-

FIGURE 1. Effective compression is the cornerstone of large-scale heat pump installations. Integrally-geared compressors are often deployed in such applications

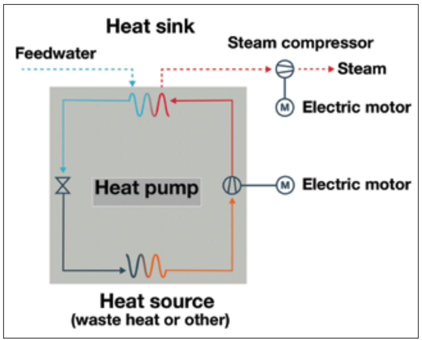


FIGURE 2. Steam can be produced using a heat pump setup that may include additional steam-compression capacity

lowed by a compressor. The working fluid, once heated and compressed, is passed to a condenser located at the heat sink where the feedwater is evaporated into steam. Finally, a throttle valve or expander completes the closed-loop heat pump cycle. Depending on the process steam conditions — and the refrigerant and specific heat pump configuration used — a simple heat pump cycle can be installed where required steam conditions are below 2 bars, given that simple high-temperature cycles are limited to around 150°C.

In cases where the process demands higher steam pressures, a secondary compressor stage can be added, either integrated into the heat pump compressor or installed as a separate motor-driven unit (Figure 2). Many industrial heat applications require multi-stage compression, which can be achieved by integrally geared compressors. As with many aspects of heat pump technology, integrally geared compressors are a proven technology with millions of hours of service across a substantial spread of industrial applications. Steam compressors deployed in conjunction with a high-temperature heat pump can see steam conditions reach temperatures of up to 300°C and pressures of 60 bars. A wide range of process steam requirements can thus be generated using a steam-production heat pump (Figure 3).

Steam pressure between the heat pump and the steam compressor can be varied to achieve the best overall efficiency. Lower interim pressures are generally associated with better efficiency, but do require a larger steam-compression component. Actively cooling the steam during the compression phase by injecting water may also be used to boost overall efficiency and also allows a smaller heat pump to be employed in producing process steam. In any event, the combination of heat pump and steam compressor typically yields a COP ranging from just under 2 to over 3, depending on the refrigerant and the cycle configuration.

The inherent flexibility of the heat pump system gives it ample scope to support the cost-effective decarbonization of industrial sectors that are typically considered as particularly challenging targets. Indeed, power-to-heat technologies are the most efficient approach to achieving decarbonization goals, specifically through the integration of industrial high-temperature heat pumps. In particular, the compressor heat pump can be

powered directly using renewable energy, making any heating or cooling capacity essentially carbon-free. Many industrial sites have the scope for onsite renewables or can sign contracts for the supply of low-carbon renewable electricity. Furthermore, the heat pump approach allows the effective use of both waste heat and ambient sources of energy. Heat pumps can thus deliver high-pressure steam by recovering process energy from cooling water or other process streams. Many industrial sites are also located adjacent to large bodies of water, making that element of heat pump deployment also feasible.

Heat pumps in play

Heat pumps are not a new concept for the CPI and are already commonly used in some processes. For example, during propylene production, heat pumps have been used for many years in the recovery and purification phase of the propane dehydrogenation (PDH) process.

High-temperature heat pumps clearly offer a proven way to convert renewable electricity and low-quality heat sources into useful high-value process heat and steam for industrial applications. This approach delivers multiple benefits — in particular a route to both decarbonizing process heat and electrifying heat production. These qualities displace fossil fuels, in turn reducing primary energy costs, as well as improving the security of energy supply and avoiding extreme price volatility. These are factors that have characterized the energy market over recent years. Improved energy efficiency in industrial processes not only leads to substantial primary energy savings and subsequent reduction of CO_2 emissions, though. Much renewable generation capacity is variable, given its derivation from wind and solar energy. During periods of surplus renewable energy, heat pumps can be adjusted to draw additional power and effectively soak up this excess. This improves the stability of the grid and helps operators maintain voltage and frequency control. These faculties potentially represent another revenue stream for heat pump operators. Similarly, heat pump operations may be reduced during periods when renewable-energy output is low in comparison with demand. In each case, the heat storage capacity of the system must be considered, but these novel operational modes are certainly feasible.

A sound business decision

Medium- and larger-sized plants offer a significant potential for large high-temperature heat pump deployments, and are good candidates for heat pump use because of the large decarbonization potential available in the CPI. However, heat pumps inevitably face competition from the well-established fossil-fuel heating systems currently used in the CPI. Despite their environmental advantages and proven capabilities, heat pumps understandably face some skepticism regarding their financial viability. A cost comparison between heat pumps and conventional boilers effectively dispels any concerns, given the cost of fossil fuels and their associated carbon emissions certificates, which are major cost considerations.

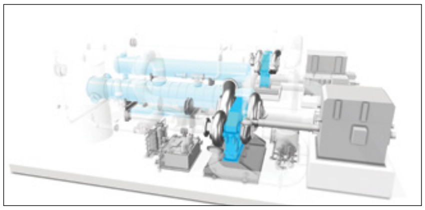


FIGURE 3. This figure shows the typical arrangement of a heat pump with an additional steam compressor, which can allow the system to achieve higher steam temperatures and pressures

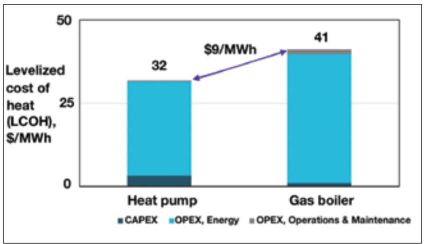


FIGURE 4. The LCOH (steam production) is compared between a heat pump and gas-fired boilers. The basis for this comparison assumes an estimated electricity price of $80/MWh and a gas price of $35.31/MWh

For example, a heat pump system using ammonia as a working fluid could generate 85 ton/h of superheated steam at 3.5 bars and 200°C using a cooling water circuit in a petrochemical production process as a heat source with a temperature of around 60°C. This configuration can achieve a COP of 2.8. Under these conditions, the heat pump would run for 8,500 h/yr for its 30-yr estimated lifespan. Although the capital expenditure (CAPEX) for a heat pump system is higher, by a factor of about three, the operational expenditure (OPEX) is far lower than for a gas-fired boiler. In this scenario, the return on investment for the heat pump is around 5.5 years, while a levelized cost of electricity (LCOE) comparison makes the heat pump solution a solid business decision in comparison with a fossil-fueled alternative (Figure 4). Heat pumps are certainly highly efficient — for the supply of process-steam, they can achieve a COP of up to 3. For reference, an electric boiler would typically have a COP of less than 1.

Given the ability of heat pumps to offer heat (as hot water or steam) or cooling for multiple processes, it is an approach well suited to numerous industrial sectors. This is especially the case as many industries are making substantial efforts to reduce their direct and indirect greenhouse gas emissions. The route to a net zero industry will rely on improving efficiency and limiting the need for fossil-derived energy wherever possible. Operating boilers with low-carbon fuels like biogas or green hydrogen is one approach, but new process technologies that enable renewable-driven power-to-heat capabilities offers far more scope. ■

Edited by Mary Page Bailey

All images provided by MAN Energy Solutions

Author

Jörg Freckmann is a senior manager in strategic business development for heat pumps, energy storage and petrochemical applications at MAN Energy Solutions (joerg.freckmann@man-es.com). Prior to his current role, he spent several years working as sales manager for gas and steam turbines for power generation applications across many industry sectors, including pulp and paper, waste-to-energy, biomass, concentrated solar power plants and mechanical drive applications in the chemical and petrochemical industries. He studied mechanical engineering at the Ruhr University Bochum, Germany, specializing in power generation, turbomachinery and gas and steam turbines.

The Ten Most Common Laboratory Ventilation Mistakes

A laboratory ventilation system that does not work effectively may not keep personnel safe and can produce significant adverse impacts on operations

Richard P. Palluzi
Richard P Palluzi LLC

Laboratory ventilation is very different from commercial and industrial systems. As a result, many experienced heating, ventilation, air conditioning (HVAC) engineers, designers and contractors can go through their entire careers without having to design a laboratory system, so they are much less familiar with a laboratory's specific requirements.

Laboratory personnel treat their ventilation systems as a given. They expect them to be designed right, to always work properly, and to require no real effort or understanding on their part. Most end users have only the most rudimentary understanding of how their own systems work, alarm, and can fail. Yet they implicitly rely on these systems to keep them safe.

The convergence of these two factors often results in laboratory ventilation systems that are poorly designed, marginally functional and not as safe as assumed.

Laboratory exhaust ventilation is expensive. Estimates range from \$20–70/(ft^3/min) to install and \$3–12/(ft^3/min) to operate depending on local costs and system design. Hoods (Figure 1) are an added expense, ranging from \$200–400 per linear foot to purchase and install. Hood controls add another \$1,000-\$3,000 per hood. This means it is not uncommon for ventilation systems to account for 15–40% of the total cost of a new laboratory. Hence, it is not surprising that designers, contractors and management all want to minimize the amount of exhaust provided to lower the capital costs of a new or modified laboratory.

The trick to minimizing these costs while ensuring safety and operability is to avoid these common mistakes.

FIGURE 1. The hood is a common site in all laboratories, from schools (shown here) to industrial sites

Ten common mistakes

1. Lack of adequate laboratory exhaust to meet operational requirements. A laboratory is first constructed based on a conceptual design with an associated budget and schedule. Too often this conceptual design fails to address all the laboratory exhaust requirements. Many exhaust requirements need a time-consuming and detailed survey to identify; some will even require taking actual measurements. Neither is something usually amenable to the schedule and resource constraints of a conceptual estimate.

Another issue is that some installations may have too little ventilation for their current (and future needs). This requires a careful evaluation by the organization's safety personnel to identify and articulate their revised basis, another time-consuming and resource-intensive task. As a result, conceptual exhaust requirements, and their attendant costs, are almost always underestimated — often badly.

The best way to avoid this issue is to provide adequate exhaust contingency in the initial scoping design and cost. Contingency is an allowance for historically predictable but currently unknown factors. While contingency is routinely applied to cost estimates, commonly but less frequently to schedules, it is rarely if ever applied to conceptual exhaust requirements. As a result, the need for several local exhausts not identified before, that extra hood (or more often hoods) that got missed, the new hood for a new program, and routine growth all combine to make almost every laboratory short of exhaust before it is even built.

Compounding this problem is that ventilation is the major expense for a new laboratory. So, when budgets run tight, the first place value engineering (the politically correct term for cost cutting) always starts is by trying to cut some ventilation. The result of these efforts is a laboratory that does not have the exhaust it needs to work properly. This problem sometimes manifests itself as soon as the new laboratory is built; it almost always surfaces within a few years. The organization must ensure that enough ventilation is included in all stages of the project. Typically, I recommend at least 20% extra exhaust be added as contingency in the conceptual design stage to try and account for these factors.

2. Too optimistic an assumed diversity factor. The diversity factor is the number of hoods or other exhaust systems likely to be operational at any one time. A 70% diversity factor assumes that no more than 70% of all the hoods or other exhausts on a fan will ever be in use

FIGURE 2. Installing a mechanical stop or warning label at the maximum height of the hood sash is not a reliable way to limit the hood opening

at any one time. It is used to size the capacity of modern variable exhaust systems. These systems automatically vary the speed of the fan to match the currently in use hood exhaust needs. As more hoods are opened for use, the fan speeds up. As hoods are closed and not in-use, the fan slows down.

These systems, while costly, pay for themselves within a few years through energy savings. If the design diversity is ever exceeded, the fan cannot supply enough exhaust and so hood performance will begin to suffer and personnel may be exposed — unknowingly — to hazardous materials.

A common problem is developing a diversity factor based on either general guidelines, cursory surveys, limited experience at other facilities, or limited monitoring. It is very easy to compile data that show 50% of all hoods are not in use over the course of a year. However, it requires more effort to recognize that these data are skewed by holidays, vacations and similar general periods of lower use, which obscures the fact that on many days, the actual use will reach or exceed 70%. Diversity has to always be more than is ever needed, rather than just the average.

Other issues can acerbate this problem. Are hood doors routinely left open when not in use? This will increase the apparent exhaust requirements and required diversity factor. Conversely, a design based on a reduction in exhaust assuming all hoods will always be fully closed when not in use even for a moment is not realistic, as people will not always comply. More use (during a growth phase or unusually busy period) or less use (during a downturn or slow period) skews the results. Surveys are often notoriously inaccurate as they are based on people's perceptions, which are often very different from the reality, or affect the outcome by the very act of looking at it. (Walking into a laboratory and mentioning one is looking at hood use is very likely to result in the occupants deciding to await one's departure before doing anything.)

When the diversity factor is breached, meaning more hoods are in use at a given time than the system can support, there is no easy fix and safety is likely to be compromised. Most laboratories, when asked how they know this breaching of the diversity factor has occurred and what is their plan to deal with it, respond with a resounding silence. And a tight budget promotes a downward diversity estimate with often attendant problems later.

3. Limiting hood opening sizes. Designers suggest limiting hood openings to save capital and operating costs. The most common reason is that the cost of the laboratory has been underestimated. When the design is complete and the costs have risen above the budget, the design firm is stuck with trying to find ways to reduce the costs back to within budget. The easiest way to do this is to reduce the size of the laboratory or reduce the size of the ventilation system. Owners accept the latter suggestion as it is much more palatable at that stage of the project to agree to limit the hood opening than to explain to everyone that they must reduce their laboratory space and/or the number of hoods they will have. The owners convince themselves that they can come up with a procedure their folks will follow and make it workable without any real operational impact.

Less often, but still often enough, the problem arises in an established facility that has run out of exhaust due to modifications and new installations. In this case, the designer's options are very few. Either spend a large amount of money and time, and create significant disruption to expand the ventilation system or get agreement to limit the hood openings. Again, the owner feels that the easiest solution is to limit the size of the hood opening as the best of only awful options.

The most common approach is to install a mechanical stop or warning label at the maximum height the hood sash is designed to be open (Figure 2). Neither approach works in real life. Mechanical stops are modified or removed. Labels are ignored or removed. Almost every survey I have ever conducted, even when they know I am coming, shows numerous sashes open well above their design limits. Explaining the potential hazards to operating personnel rarely corrects the problem for more than a few days, at most. In many cases, the maximum design openings are so small as to realistically make normal operations cumbersome to impossible. Hence, operating personnel must ignore them to get their work done.

4. Lack of adequate supply air. All researchers eventually require more exhaust for new equipment or operations. Providing this exhaust is always expensive so many design firms will not provide the matching amount of supply air to feed the exhaust. (Doing so effectively doubles

FIGURE 3. Ventilated enclosures fail to capture effectively as they do not have a properly designed plenum to distribute the exhaust

FIGURE 4. Local exhaust can be a great mitigative measure when properly designed, installed and properly used

the costs.) As a result, over time, many (I dare say most) laboratories become much too negative as the total exhaust significantly exceeds the total supply. This means the exhaust is working harder to find the air to exhaust because it is too constricted. As a result, a fan that should be able to exhaust 1,000 ft^3/min may only exhaust 900 ft^3/min or less. This adversely reduces hood effectiveness, makes passing a hood face velocity test more difficult, often draws air in from other laboratories that are not as negative, and makes it harder to open doors. In fact, some laboratories get so negative that they waste a significant amount of the new exhaust they so expensively added.

The problem is that the effect of a 1% supply shortfall is almost always acceptable. However, if the 1% shortfall is actually 2%, and if the process is repeated several times so that the total shortfall is now 10% or more, or if the design basis fails to recognize where the shortfall will be drawn from, major operating problems can — and usually do — arise. Any increase in exhaust must be matched to an increase in supply to be effective.

5. Assuming alternative ventilation approaches will perform as effectively and safely as a hood. Purchasing and installing hoods and their attendant supply and exhaust systems are so expensive, there is a tendency to try and move some operations from a capital-intensive hood to less costly alternatives. These may include ductless hoods, ventilated enclosures, canopy hoods, laminar-flow cabinets and other alternatives. While all these devices may be effective if a hazard analysis and risk assessment shows that they are suitable for the envisioned operations and if they are properly selected, designed, installed and used, few meet all these requirements. Worse, none of them will perform as well as a hood in all operations all the time.

Ventilated enclosures (Figure 3) are fabricated with no real design work and fail to capture effectively because they do not have a properly designed plenum to distribute the exhaust. Ductless hoods can work effectively for specific uses but require procedures to confine their use to the design basis and require routine costly filter changes. Canopy hoods fail to capture all but the lightest gases under the most optimum conditions and usually are useless. Laminar-flow hoods, useful for solids handling, are used for the same operations as regular hoods with gases and liquids and fail to work effectively due to a face velocity that is too low. All these issues revolve around a failure to thoroughly analyze the required operations and evaluate what mitigative measures are suitable for the risks involved.

The National Fire Protection Association (NFPA) standard NFPA 45 Fire Protection for Laboratories Using Chemicals clearly warns against using any of these alternatives as a replacement for a standard hood without careful analysis. A detailed hazard analysis and risk assessment usually ends up with an assessment that "we really need a hood." Instead, the specter of going over budget often drives research organizations to incredibly poor decisions based on naïve hopes that these less expensive options will work adequately. Usually, they create long-term operating problems, difficult exposure issues and problems costly to repair later.

6. Failing to understand the purpose of a hood alarm and not set-

ting it properly. A hood alarm is required by NFPA 45 Fire Protection for Laboratories Using Chemicals section 7.8.7 "for indicating that the hood airflow remains within safe design limits." It is not intended as a device that tells the operator that their average 100 ft/min face velocity is now 99 ft/min due to changes in wind against the fan exhaust, minor perturbations in a variable exhaust air system, sudden sash changes, motion near the face of the hoods or dozens of other potential reasons for a small, short-term upset. A hood alarm should only go off when the total exhaust air has fallen off significantly (at least more than 10%) for a reasonably significant period (usually a minute or more), indicating a serious problem affecting the hood's safe use has occurred. Instead hood alarms trip continually and are eventually permanently silenced, ignored or modified *Sub Rosa* (secretly) to stop going off.

7. Not maintaining a proper laboratory negative pressure or too much negative pressure. NFPA 45 7.3.3 requires all laboratories to be negative with respect to non-laboratory areas. The American Industrial Hygiene Association (AIHA) standard AIHA Z9.5 Laboratory Ventilation recommends the higher flow be from the lower, non-laboratory area to the higher hazard laboratory area in 5.2.1 unless there is no credible risk.

The most common problems I have seen involve reverse airflow, no negative pressure and excessive negative pressure. Reverse airflow almost always occurs because another nearby area is so starved of supply air that it overwhelms the laboratory's ventilation system and sucks air from an adjacent laboratory. This is a difficult problem to solve except by providing more supply air to the starved laboratory, which is often costly.

Other, less-viable solutions include requiring some doors only be used for emergency exits and providing airlocks or ante chambers. Other times reverse airflow is due to the supply exceeding the exhaust, pressuring the laboratory. This may be due to a design error, a poorly designed or improperly functioning control system, or a removal of exhaust within the laboratory without any corresponding adjustment to the supply. No negative pressure usually arises only when the design firm has designed the system improperly. Usually, although not always, it can be corrected by simply turning down the supply air to the laboratory. Sometimes this is not possible as the designers used the air from the laboratory to supply other areas, a practice no experienced professional would likely support. Excessive negative pressure usually arises due to a biohazard or cleanroom specification being needlessly applied to a standard laboratory. Again, turning down the supply air rate often works. Biohazard facilities and cleanrooms usually need a much higher defined air flowrate into the laboratory facility for safety, but these same requirements are often inappropriately applied to standard wet chemistry laboratories where all they do is create operating and maintenance problems and increase costs.

8. Overly complicated pressurization schemes. Both NFPA 45 and AIHA Z9.5 are clear that momentary loss of negative pressure when opening a door is acceptable. Nevertheless, many designers provide complicated systems to try and maintain the negative pressure at all times. These systems almost invariably create operating problems from too much pressure or too little pressure. A simple offset in the exhaust and supply air rates (so the supply air tracks lower than the exhaust at all times) is all that is necessary. It may be momentarily disturbed, even reversed, during a door opening or closing, but this has never been shown to be a major issue in a standard laboratory.

9. Getting too focused on total exhaust rate. Total exhaust rates for a laboratory are usually expressed in air changes per hour (ACH). NFPA 45 recommends at least 4 ACH for an unoccupied laboratory and suggests that most occupied (in use) laboratories should be over 8 ACH. The Federal Occupational Safety and Health Administration (OSHA) suggests 4–12 ACH, the American Society of Heating, Refrigerating and Air-Conditioning Engineers (ASHRAE) 6–12, the National Research Council 6–12, and the American Institute of Architects (AIA) 4–12. AIHA Z9.5 frankly notes this is "a subject of controversy."

In general, overall exhaust rates, while critical for safety during an accident or a release, are not effective in protecting the laboratory occupants from routine releases or emissions, since they will be breathing the air with the hazardous releases until it has been diluted to negligible levels. Depending on the amount released, this can be for some time. Since all operations that have a credible release of hazardous materials should take place in a hood, properly designed ventilated enclosure, sealed system, or under appropriately designed and properly used local ventilation, releases into the general laboratory environment should be rare.

NFPA 45 requires all hazardous materials be captured at the source. Spills, broken containers, momentary releases from a hood due to transfer operations, and similar minor and infrequent events are the most common causes of where the general laboratory exhaust is required. While this suggests that some level of ACH is required, it should be set more by the number of hoods, ventilated enclosures, and local exhaust the operations require rather than by an overall exhaust rate. I, personally, would be very uncomfortable with a laboratory without at least 6 ACH or, better yet, 8 ACH but higher values are not necessary. If odors are common and conditions resulting in releases prevalent, then the solution usually lies in fixing the laboratory operations or a specific issue, rather than the overall laboratory ventilation design.

10. Relying on local exhausts to capture vapors and limit exposures. Local exhaust (Figure 4) when properly designed, installed and — most importantly — properly used is a great mitigative measure. To work properly, the local exhaust must be positioned within the design basis. This basis is often much

closer to the work that is releasing vapors than is operationally desirable. If the distance from the release is increased, significantly more exhaust is required or effectiveness becomes substantially worse. All too often, the design economics dictate an exhaust that needs to be within a few inches above the top of the operation. This position is often inconvenient and invariably ends up being used 2–3 times further away with attendant significant loss in effectiveness. Worse, most local exhausts are often simply flexible hoses to lower costs. To position these properly requires a third hand, second person, or some sort of jury rigged holder. The first is sadly not a part of human anatomy, the second rarely available, and the last usually much further away than what the exhaust was designed for. Most, although not all, operations that rely on a local exhaust really need to take place in a hood.

Final remarks

What is the bottom line of all these common recurring problems? A laboratory ventilation system that does not work effectively may not keep personnel safe or can produce significant adverse impacts on operations. I strongly suggest you review your ventilation design very carefully before signing off on the design. This is particularly important if the designer or design firm is less experienced in laboratory ventilation. (And many who claim to be, sadly are not.) I suggest that a cold-eyes review by a laboratory ventilation specialist is often the best money you can spend in a project, because correcting most of these issues after construction ranges from the very expensive to the impossible. ■

Edited by Gerald Ondrey

Note: All photos courtesy of the author, unless otherwise indicated.

Author

Richard P. Palluzi of Richard P Palluzi LLC (72 Summit Drive, Basking Ridge, NJ 07920; Phone: 908-285-3782; Email: rpalluzi@verizon.net) is a consultant to the pilot plant and laboratory research community on safety, design and research project management. He retired as a Distinguished Engineering Associate after 40 years at ExxonMobil Research and Engineering, where he was involved in the design, construction and support of pilot plants and laboratories for ExxonMobil affiliates worldwide. Palluzi is the author of two books, over 180 articles and 60 presentations. He was also one of the core team contributors to CCPS's new book, "Handbook For Process Safety In Laboratories And Pilot Plants, A Risk-Based Approach." He has chaired the AIChE Pilot Plant Committee, ExxonMobil's Pilot Plant and Laboratory Safety Standards Committee, and ExxonMobil's Clinton site Safe Operation Team. Palluzi is a member of AIChE's Process Development Division and was elected an AIChE Fellow in 2021. He is on the Technical Committees for National Fire Protection Association NFPA-45 Fire Protection for Laboratories Using Chemicals, NFPA-55 Industrial and Medical Gases, NFPA-2 Hydrogen Technologies Code, and Z9.5 Laboratory Ventilation. He has a BE(ChE) and an ME(ChE) from Stevens Institute of Technology and is a licensed Professional Engineer (PE) and Certified Safety Professional (CSP). Palluzi now consults full time on all aspects of safety, design, and operation for pilot plants, laboratories and research facilities. He also develops and teaches numerous public and custom training on pilot plants and laboratories.

A Brief Review of Natural Water's Influence on Scale Formation in Heat Exchangers

Understanding the water chemistry is a first step in preventing heat-exchanger fouling

Brad Buecker
Buecker & Associates

At the two power plants and chemical plant in which this author formerly worked, the freshwater-makeup supplies (two lakes and an underground aquifer adjacent to a river) all had a consistent pH at or slightly above 8.0. This pH range is common for many surface supplies, and typically comes from bicarbonate alkalinity (HCO_3^-) that naturally dissolves in these water bodies. But from where does this alkalinity arise? Per the excellent discussion of this subject in Ref. 1, we briefly explore this issue and also examine what can happen in heat exchangers without proper chemistry control.

Geology is one key

One of the most common mineral deposits near the earth's surface is limestone, whose principal component is calcium carbonate ($CaCO_3$). This versatile mineral serves as a raw material for numerous important industrial and infrastructure applications including concrete, water-treatment chemicals, scrubbing reagent, and simply for gravel roads.

Many surface waters are in contact with limestone formations, and groundwaters often percolate through limestone and settle in aquifers that are bounded by mineral. Calcium carbonate has a strong crystal lattice, and thus $CaCO_3$ is only slightly soluble in water.

$$CaCO_3 \leftrightarrows Ca^{2+} + CO_3^{2-} \quad \textbf{(1)}$$

$$K_{sp}(25°C) = [Ca^{2+}][CO_3^{2-}] = 4.6 \times 10^{-9}$$

Per this solubility product, the molar concentrations of calcium and carbonate in neutral water would be 6.8×10^{-5}, which is indeed very slight. But an additional factor must be considered. CO_3^{2-} is a fairly strong base and will hydrolyze water to some extent.

$$CO_3^{2-} + H_2O \leftrightarrows HCO_3^- + OH^- \quad \textbf{(2)}$$

Combining Equations (1) and (2) shows the overall reaction of $CaCO_3$ in neutral water.

$$CaCO_3 + H_2O \leftrightarrows Ca^{2+} + HCO_3^- + OH^- \quad \textbf{(3)}$$

Calculations indicate that CO_3^{2-} hydrolysis of water increases the limestone solubility from 6.8×10^{-5} M at 25°C to 9.9×10^{-5} M. An important point to keep in mind is that these reactions produce hydroxide alkalinity (OH^-), even if only in slight concentrations.

Now, let's look at the other end of the spectrum and a primary reaction that makes natural waters non-neutral and which greatly influences chemistry.

Atmospheric influences

Natural waters absorb carbon dioxide from the atmosphere. While it is often argued that much of the CO_2 exists as hydrated molecules, the following equations sufficiently represent the chemistry.

$$CO_2 + H_2O \leftrightarrows H_2CO_3 \quad \textbf{(4)}$$

$$H_2CO_3 \leftrightarrows H^+ + HCO_3^- \quad \textbf{(5)}$$

The lowest pH in natural surface waters that can be achieved by these reactions (excluding acid rain issues) is around 5.6, but the solution is still acidic, which is very important. Consider again, Equation (3). When the acidity generated by CO_2 absorption interacts with the alkalinity generated by the fractional $CaCO_3$ dissolution, the hydrogen and hydroxyl ions combine to form water, and per Le Chatelier's Principle, Reactions (3) and (5) are both driven to the right. This synergistic effect can produce water with a HCO_3^- concentration of 1×10^{-3} M (equivalent to about 60 parts per million as the species), and "a pH of about 8.3" [*1*]. The relationship of the carbonate species is clearly illustrated in Figure 1.

This same acid-base synergy is what makes high-purity limestone (high $CaCO_3$ content) quite reactive and economical as a scrubbing agent, when ground to very fine particles, in wet fluegas desulfurization systems. Aqueous sulfur dioxide (SO_2) is a stronger acid than CO_2, and analytical data have shown nearly complete $CaCO_3$ reactivity in well-designed scrubbers [*2*].

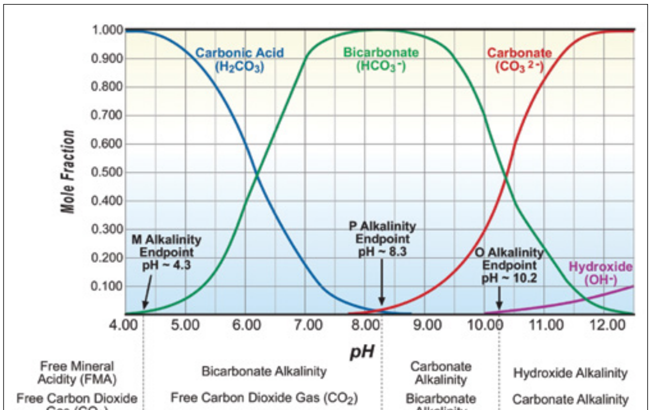


FIGURE 1. The relationship between carbonate species in water is shown here. For waters that pass along or through limestone deposits and that absorb CO_2 from the atmosphere, the reactions are driven towards the maximum HCO_3^- alkalinity

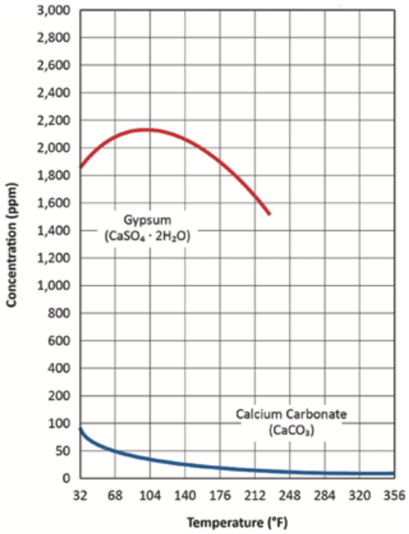


FIGURE 2. This graph shows the inverse solubility of two of the most common minerals in natural waters

FIGURE 3. Shown here is $CaCO_3$ scale in an extracted and bisected heat exchanger tube

Carbonate chemistry in reverse

Now let's briefly examine some of the above chemistry in reverse, and show how it can influence equipment operation and performance at facilities in the chemical process industries (CPI).

From the time humans began heating water for cooking and sanitary purposes, they have undoubtedly observed mineral deposition in heated vessels. These issues became acute following the invention and expanding use of steam engines during the Industrial Revolution of the 18th and 19th centuries. The primary culprit is calcium carbonate.

$$Ca^{2+} + 2HCO_3^- + \text{heat} \leftrightarrows CaCO_3\downarrow + CO_2 + H_2O \quad (6)$$

A key aspect of this chemistry is the inverse solubility of $CaCO_3$, as illustrated in Figures 2 and 3.

Accordingly, in many systems that have cooling towers for primary heat exchange, the cycling up in concentration of dissolved elements and compounds in the cooling water, combined with the heat increase in

TABLE 1. OTHER COMMON COOLING-WATER SCALE DEPOSITS (NOT EXCLUSIVE)

Compound	Formula
Gypsum	$CaSO_4 \cdot 2H_2O$
Silica	SiO_2
Magnesium Silicate	$MgSiO_3$
Calcium Phosphate	$Ca_3(PO_4)_2$
Fluorite	CaF_2

various types and styles of heat exchangers, can lead to serious scale formation. This article highlights $CaCO_3$, which is usually the first deposit to form in untreated or poorly treated cooling water, but other well-known and problematic deposits include, but are not limited to, those shown in Table 1.

And now, as industrial plant personnel, either by choice or mandate, switch from fresh-water supplies to alternatives, such as municipal wastewater-treatment plant effluent, proper scale (and corrosion) control methods have become even more critical. Additional details are available in Ref. 3. ■

Edited by Gerald Ondrey

References

1. Baird, C., "Environmental Chemistry," Second Edition, W.H. Freeman and Company, New York, N.Y., 1999.
2. Buecker, B., Wet Limestone FGD Solids Analysis by Thermogravimetry, proceedings of the 24th Annual Electric Utility Chemistry Workshop, May 11–13, 2004, Champaign, Illinois.
3. Buecker, B., (Tech. Ed.), "Water Essentials Handbook"; 2023. ChemTreat, Inc., Glen Allen, Va., available as a downloadable eBook at www.chemtreat.com.

Acknowledgment

All figures courtesy of ChemTreat, Inc.

Author

Brad Buecker is president of Buecker & Associates, (Lawrence, Kan.; beakertoo@aol.com) specializing in technical writing and consulting. Most recently, he served as senior technical publicist with ChemTreat, Inc. Buecker has over four decades of experience in or affiliated with the power and water-treatment industries, much of it in steam generation chemistry, water treatment, air quality control, and results engineering positions with City Water, Light & Power (Springfield, Illinois) and Kansas City Power & Light Company's (now Evergy) La Cygne, Kansas station. He also spent two years as acting water/wastewater supervisor at a chemical plant, and eleven years with two engineering firms, Burns & McDonnell and Kiewit. Buecker has a B.S. in chemistry from Iowa State University with additional course work in fluid mechanics, energy and materials balances, and advanced inorganic chemistry. He has authored or co-authored over 250 articles for various technical trade magazines, and has written three books on power plant chemistry and air pollution control. He is a member of the ACS, AIChE, AIST, ASME, AWT and the Electric Utility Chemistry Workshop planning committee.

Complex Chemistry Demands Precise Process Control

An advanced control system helps alleviate workflow inefficiencies and reliance on manual adjustments in a complex chemical process

Damas Limoge and Cecilia McLaren
Nanotronics

Synthesizing principles of engineering and biology, Solugen Inc. (Houston; www.solugen.com) has developed and commercially deployed a unique carbon-negative platform for peroxide manufacturing. The platform, called Bioforge, employs a renewable plant-based extract and combines it with deionized water and compressed air, which is then processed through an enzyme reactor to continually extract molecules as the reaction is occurring. The intermediate molecules are imbued with more compressed air and poured over Solugen's metal catalysts to create a final product. For more details on Solugen's process, see Enzyme Engineering Enables Bio-based Hydrogen Peroxide, *Chem. Eng.*, May 2019, p. 7.

The molecules Solugen produces comprise the foundational building blocks of ingredients in numerous manufacturing sectors, including agriculture, water treatment, oil-and-gas, cleaning products, food additives, personal care and concrete production. Solugen has a location in Slaton, Tex. and a primary manufacturing plant in Houston (Figure 1). As with any new process technology, commercialization and subsequent stable operation involve many technological hurdles. This article describes the implementation of an advanced process-control scheme that enabled Solugen to reduce operational inefficiencies and improve production yield.

FIGURE 1. At Solugen's manufacturing plant in Houston, a novel technology uses enzymatic reactors to produce carbon-negative chemical building blocks

Overcome scalability challenges

Like any new process, scalability is a concern. Within Solugen's process, there is critical variation across the catalysts that are deployed and the chemistry on which the process relies. This variation requires quasi-human interpretation of a given state to successfully progress the process. Through continuous use, the particles become inconsistent in structure and do not perform in agreement with the theoretical ideal. Therefore, operators must be able to adjust plant settings to account for discrepancies in saturation, which can be unreliable when left to human capacity or inference alone. Introducing a deep-learning control strategy to the plant was seen as one potential solution for real-time optimization of such complex objectives. To adjust for variation in particle composition, it is necessary to derive an intelligent control scheme that automatically adapts to process fluctuations *in-situ*.

Nanotronics (Cuyahoga Falls, Ohio; www.nanotronics.co) was consulted by Solugen to test the applicability of the nControl proprietary process-control platform. The nControl system builds latent representations of critical information using empirical observations in the form of sensor data, extracting features contributing to deviation in normal performance. It is able to surmise latent variables, whose analytical definition is nearly intractable, and provide a rich snapshot of the past, present and future state of the process.

The nControl prediction and control models operate through dimensionality reduction — compressing informationally sparse states, or data extracted from disparate sensors, into a representation that conserves important attributes of the observed phenomenon. This results in higher informational density for both humans and machines to make operational decisions — a key benefit in Solugen's process, where human interaction is classically the first response to varying process parameters.

Measurements are encoded with nonlinear, time-aware and sensor-agnostic deep networks, ensuring that each transformation embeds additional, pertinent information in its latent representation. These representations are simultaneously fed forward for the ultimate purpose of control via artificial intelligence (AI), but are also interpreted for immediately useful consumption by human observers, in the form of anomaly detection and alerting, as well as quality metrics.

The anomaly detection capabilities nControl offered to Solugen were highly effective in streamlining and refining the site's chemical processes. The platform is able to adjust for, as well as predict, degradation of capital equipment, unanticipated restriction of

FIGURE 2. The nControl platform provides process adjustments and predictions related to degradation of equipment, such as valves and pipelines, within Solugen's plant

the valves and pipes that house feedstocks and atypical chemistry correlated to exothermic events. Inferred quality metrics allow engineers and operators to determine and execute goals in real time by providing immediate feedback on process deviations with acute accuracy (Figure 2).

Solugen's control strategies benefit from a reduction in the dimensionality of inferred quality metrics that highlight the important features of both human-legible, useful observations, and the values targeted by the control strategy, which is able to act faster and more precisely than a human agent.

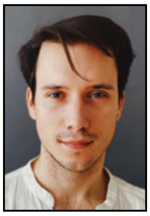
Such dynamic performance is unique among control systems for complex chemical processes. Competing platforms operate as backward-looking manufacturing analytics that do not include real-time process control. They may provide retrospective reports and insights or instantaneous monitoring and alerting, but they do not have the capacity to actively control production processes, and no software-only solution is capable of making these interventions. Typically, factory operations must be networked via a programmable logic controller (PLC) to achieve this effect, whereas the nControl platform can act on occurring processes immediately.

Furthermore, nControl is nonparametric, meaning that it learns to recognize new empirical patterns not captured in theoretical formulations of the production process, such as physics-based or chemistry-based simulations. It is also inherently multivariate and retains interactions between sensors. Its multiscale structure enables it to capture periodicities and other patterns at different time scales — predictions can be thought of as a composite of multiple timelines.

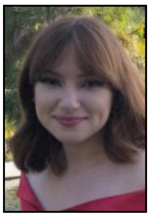
For Solugen, the initial models from Nanotronics are unsupervised and do not require labor-intensive data labeling. The system deftly handles datasets with significant variance and can be trained on a variety of operations — startup, shutdown, maintenance and various run conditions — and has successfully predicted processes across diverse situations, including conditions that were not visible in training data. ■

Edited by Mary Page Bailey

Authors



Damas Limoge is the director of applied research at Nanotronics (2251 Front Street, Suites 108-109, Cuyahoga Falls, OH 44221; Phone: 330-926-9809; Email: dlimoge@nanotronics.co), focusing on nonlinear system control, real-time analysis with generative autoencoders, time-series attention networks, deep reinforcement learning techniques and developing the nControl and AIPC platforms. He holds a M.S. degree in mechanical engineering from the Massachusetts Institute of Technology (MIT) and has previously worked with MIT's Lincoln Laboratory, Resolute Marine Energy, General Electric and Goodrich in various mechanical engineering and simulation design roles.



Cecilia McLaren is a marketing and communications associate at Nanotronics (Brooklyn Navy Yard Building 20, Brooklyn, NY 11205), where she serves as a writer, editor and media producer. She is the producer of Utility + Function, Nanotronics CEO Matthew Putman's podcast, which explores an interdisciplinary range of subjects, including AI's impact on social, economic and political spheres. McLaren holds a B.S. degree in media, culture and communication from New York University, and has been involved with the university's Digital Theory Lab, a research cohort convening graduate students and faculty members from universities across the globe to conduct multimodal inquiry converging technological artifacts and humanities-based methodologies.

Note: nControl is a registered trademark of Nanotronics

Advertisers Index

See bottom of opposite page for advertising sales representatives' contact information

Classified Index February 2024

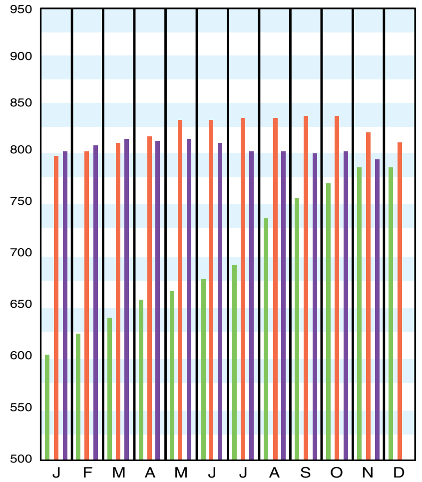
FOR ADDITIONAL NEWS AS IT DEVELOPS, PLEASE VISIT WWW.CHEMENGONLINE.COM

February 2024; VOL. 131; NO. 2

Economic Indicators

2021 2022 2023

Download the CEPCI two weeks sooner at www.chemengonline.com/pci



CHEMICAL ENGINEERING PLANT COST INDEX (CEPCI)

(1957–59 = 100)	Nov. '23 Prelim.	Oct. '23 Final	Nov. '22 Final
CE Index	789.2	790.7	814.6
Equipment	990.3	992.2	1,033.2
Heat exchangers & tanks	804.9	808.1	861.7
Process machinery	1,014.7	1,016.0	1,041.1
Pipe, valves & fittings	1,331.4	1,330.7	1,461.7
Process instruments	560.5	560.8	555.6
Pumps & compressors	1,484.5	1,484.4	1,323.1
Electrical equipment	804.4	802.6	785.7
Structural supports & misc.	1,096.4	1,103.5	1,153.7
Construction labor	373.7	374.2	359.1
Buildings	797.2	800.9	802.4
Engineering & supervision	315.1	315.1	311.8

Annual Index:

2015 = 556.8

2016 = 541.7

2017 = 567.5

2018 = 603.1

2019 = 607.5

2020 = 596.2

2021 = 708.8

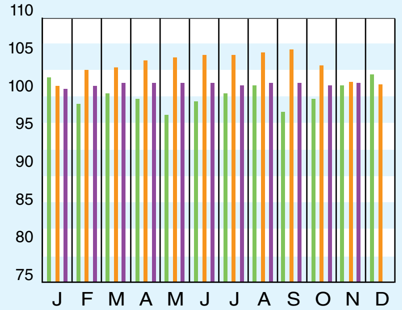
2022 = 816.0

Starting in April 2007, several data series for labor and compressors were converted to accommodate series IDs discontinued by the U.S. Bureau of Labor Statistics (BLS). Starting in March 2018, the data series for chemical industry special machinery was replaced because the series was discontinued by BLS (see *Chem. Eng.*, April 2018, p. 76–77.)

CURRENT BUSINESS INDICATORS

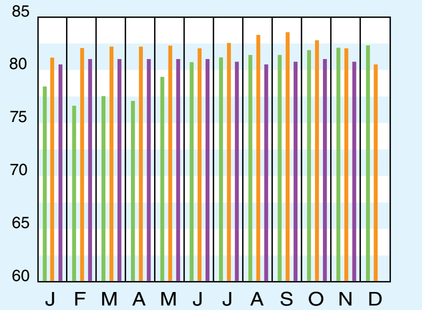
	LATEST			PREVIOUS						YEAR AGO		
CPI output index (2017 = 100)	Nov. '23	=	99.1	Oct. '23	=	99.4	Sept. '23	=	100.2	Nov. '22	=	99.8
CPI value of output, $ billions	Nov. '23	=	2,397.7	Oct. '23	=	2,398.4	Sept. '23	=	2,464.9	Nov. '22	=	2,483.8
CPI operating rate, %	Nov. '23	=	78.7	Oct. '23	=	79.1	Sept. '23	=	79.7	Nov. '22	=	80.1
Producer prices, industrial chemicals (1982 = 100)	Nov. '23	=	308.9	Oct. '23	=	315.9	Sept. '23	=	309.2	Nov. '22	=	341.7
Industrial Production in Manufacturing (2017 =100)*	Nov. '23	=	99.2	Oct. '23	=	98.8	Sept. '23	=	99.6	Nov. '22	=	100.0
Hourly earnings index, chemical & allied products (1992 = 100)	Nov. '23	=	228.9	Oct. '23	=	224.0	Sept. '23	=	227.4	Nov. '22	=	209.5
Productivity index, chemicals & allied products (1992 = 100)	Nov. '23	=	90.5	Oct. '23	=	90.8	Sept. '23	=	91.7	Nov. '22	=	90.2

CPI OUTPUT INDEX (2017 = 100)†



CPI OUTPUT VALUE ($ BILLIONS)

CPI OPERATING RATE (%)



*Due to discontinuance, the Index of Industrial Activity has been replaced by the Industrial Production in Manufacturing index from the U.S. Federal Reserve Board.
†For the current month's CPI output index values, the base year was changed from 2012 to 2017
Current business indicators provided by Global Insight, Inc., Lexington, Mass.

CURRENT TRENDS

The preliminary value for the *CE* Plant Cost Index (CEPCI; top) for November 2023 (most recent available) is down by a small margin from the previous month, continuing a string of monthly declines since June 2023. In November, small decreases were observed in the Equipment, Buildings and Construction Labor subindices, while the Engineering & Supervision subindex remained the same. The current CEPCI value now sits at 3.1% lower than the corresponding value from November 2022. Meanwhile, the Current Business Indicators (middle) show small decreases in the CPI output index, the CPI operating rate and the CPI value of output for November 2023, as well as a decrease in producer prices for industrial chemicals.